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An Interlaboratory Evaluation of the 1980 Version of the National Bureau of Standards Test Method for Assessing the Acute Inhalation Toxicity of Combustion Products

U.S. DEPARTMENT OF COMMERCE
National Bureau of Standards
National Engineering Laboratory
Center for Fire Research
Washington, DC 20234

April 1983

Final Report



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NATIONAL BUREAU OF STANDARDS

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**AN INTERLABORATORY EVALUATION OF
THE 1980 VERSION OF THE NATIONAL
BUREAU OF STANDARDS TEST METHOD
FOR ASSESSING THE ACUTE INHALATION
TOXICITY OF COMBUSTION PRODUCTS**

Barbara C. Levin, Maya Paabo, Merritt M. Birky

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U.S. DEPARTMENT OF COMMERCE, Malcolm Baldrige, *Secretary*
NATIONAL BUREAU OF STANDARDS, Ernest Ambler, *Director*

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AN INTERLABORATORY EVALUATION OF THE 1980 VERSION OF THE NATIONAL BUREAU OF
STANDARDS TEST METHOD FOR ASSESSING THE ACUTE INHALATION TOXICITY OF
COMBUSTION PRODUCTS

Barbara C. Levin, Maya Paabo and Merritt M. Birky*

Abstract

Seven laboratories selected from academia, industry, and government evaluated the 1980 version of the NBS test method for assessing the toxicity of combustion products in order to determine the operability of the procedure and the reproducibility of results across laboratories. The experimental design specified that each laboratory was responsible for testing Douglas fir and three other materials from a total of twelve natural and synthetic materials. All laboratories were required to use similar exposure and combustion systems, to measure the autoignition temperatures of their materials, to determine the toxicity of the gaseous products released by the materials under both flaming and non-flaming conditions, to monitor chamber environmental conditions (temperatures, and oxygen, carbon monoxide, and carbon dioxide concentrations), and to measure blood carboxyhemoglobin in the test animals (rats). In addition, a few laboratories measured the hydrogen cyanide generated from nitrogen-containing materials. Toxicity was evaluated on the basis of incapacitation (hind-leg flexion behavioral avoidance response) during the 30 minute exposure and of lethality during the exposure and 14 day post-exposure observation period. The results of this interlaboratory evaluation were statistically analyzed and, in most cases, demonstrated reproducible results across laboratories. Possible reasons for any inconsistencies are discussed. Sensitive experimental factors are identified and modifications to the test method which resulted from the experimental data collected during the interlaboratory evaluation are described.

Key words: carbon monoxide; carboxyhemoglobin; combustion; combustion products; hydrogen cyanide; inhalation; interlaboratory evaluation; LC₅₀; test methods; toxicity.

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1.0 INTRODUCTION

The National Bureau of Standards (NBS) has been developing a small-scale test to assess the acute inhalation toxicity of combustion products from materials thermally degraded under specified laboratory conditions. The need for this test became apparent with the realization that the majority of fire fatalities were due to smoke inhalation and not to burns. The distinct possibility that toxicants other than carbon monoxide might be present in the smoke and could be contributing significantly to the lethal atmospheres provided an impetus to design a means to evaluate these toxic atmospheres using both biological and chemical analyses.

The early studies at NBS were supported by the Products Research Committee (PRC) through a grants program designed to investigate the behavior of cellular plastics in fires [1]. As a result of the PRC-supported research and considerable input from an ad hoc working group (composed of members from academia, industry, and government knowledgeable in fire and toxicological problems), an early version of the test method was formulated in 1980 [appendix A of reference 2]. This version is the basis of this report and must be distinguished from the more recent 1982 version [3].

Nine laboratories agreed to participate in an interlaboratory evaluation (ILE) of the 1980 test method to determine the reproducibility of the experimental results. This ILE differed from a typical round robin evaluation of a test procedure in that all the laboratories did not examine all the materials at specified temperatures and concentrations. This decision was dictated by the large number of test materials (twelve), the expense of the experiments, the time necessary to test each material, and the toxicological nature of the research. According to the experimental design, each participating laboratory would examine three materials plus Douglas fir and each material would be tested by at least three laboratories. (This initial design changed, however, when two of the nine original laboratories decided not to participate in the ILE.)

¹Numbers in brackets refer to the literature references listed at the end of this report.

In addition, as the ILE progressed, the experimental results were presented at meetings of the ad hoc working group to help solve issues pertaining to the methodology of the test. This continuous input of data resulted in changes in the test method procedures. Thus the procedures being evaluated by the ILE were subject to change throughout the duration of the testing. The final result of the efforts of NBS, the ad hoc working group, and the ILE participants is the 1982 NBS version of the toxicity test method [3]. The present report describes the 1980 toxicity test method, the experimental design and the results of the ILE, the statistical analysis on the reproducibility of the data across laboratories and the subsequent modifications of the test method. When possible, this report also examines intralaboratory repeatability and sensitivity of the test method results to variations in experimental procedures and equipment.

2.0 THE NBS TOXICITY TEST METHOD

This test method was designed to assess the acute inhalation toxicity of the combustion products of materials thermally decomposed under specified laboratory conditions and was primarily intended for research and preliminary screening purposes by product researchers and material manufacturers. This test was not designed to address the problem of total toxic hazard which a fire produces or to which a specific material may contribute in a "real fire" situation although it may provide one of the input factors necessary for such a fire risk assessment. To predict the toxic hazard of a fire encompassing many materials or the added contribution by any one material to that hazard, additional factors must be considered. These factors include, among others, the amount of material and its fire properties (ignitability, rate of heat release, rate of flame spread, smoke generation, quantity of irritants present in the smoke, configuration) and the environmental conditions (ventilation conditions, presence of ignition sources, proximity of other combustibles, volume of compartments to which the smoke may spread, presence of fire protection systems, and the type of occupancy of the building). For this report, acute toxicity is defined as the harmful effects of a single short exposure (30 minutes) to combustion products generated by the thermal degradation of materials; toxic hazard is defined as the probability that a toxic atmosphere will be produced and an injury will occur as a result of the combined effect of the material's properties and environmental conditions.

A complete description of the 1980 version of this method which was the subject of the interlaboratory evaluation may be found in appendix A of reference [2]. Briefly, the test method consists of three major components: (1) a combustion system, (2) a chemical analysis system, and (3) an animal exposure system. The combustion system is a closed design in which all the combustion products are generated in a furnace located directly below the 200 liter rectangular exposure chamber (fig. 1) and are kept within the chamber except for that volume which is transferred for chemical analysis and subsequently returned. The cup furnace is similar to that designed by Potts and Lederer (fig. 2)[4]. Materials are thermally degraded at a furnace temperature 25°C above the material's autoignition temperature (flaming mode), 25°C below its autoignition temperature (non-flaming mode), and at 440°C , the temperature at which Douglas fir (the reference material in the 1980 version of the test method) was tested in the non-flaming mode. The 440°C temperature was not used if the material's autoignition temperature was less than 490°C . The autoignition temperature was determined separately for each material and was that furnace temperature which caused the material to ignite spontaneously during the 30 minute exposure period. In an actual flaming exposure, ethanol and/or an electric spark was used to ensure immediate flaming. The maximum furnace temperature for any exposure was 800°C .

Before experiments, all test materials were conditioned for at least 48 hours in a room with 40-50 percent relative humidity and a temperature of $22-24^{\circ}\text{C}$. One piece samples were tested unless the material was supplied as pellets or a powder.

The combustion products were pumped from the chamber to analytical instruments which continuously measured the concentrations of carbon monoxide (CO), carbon dioxide (CO_2), and oxygen (O_2). After analysis, all products were returned to the chamber. If continuous measurements were not possible, atmospheric samples were to be measured every five minutes for CO , CO_2 , and O_2 . In the 1980 test method, O_2 concentrations were not to fall below 18 percent. The temperature of the exposure chamber at the level of the animal's noses was also to be monitored throughout the exposure and was not to exceed 35°C .

The animals used for these experiments were male rats weighing 200-300 grams and were observed for at least 10 days prior to testing to assure the animals were healthy and acclimated to the laboratory conditions. In some laboratories, animals designated for blood analysis of carboxyhemoglobin (COHb) underwent cannulation 24 hours before experiments; this procedure involves the surgical insertion of a cannula into the animal's femoral artery [5] thereby allowing blood samples to be taken during the exposure. The blood levels of COHb are indicative of the amount of carbon monoxide inhaled by the animals. Six animals were exposed in each experiment. Each animal was placed in a restrainer (fig. 3) which was then inserted into one of the six port-holes located along the front of the exposure chamber such that only the heads of the animals were exposed to the combustion atmosphere. Exposures were for 30 minutes, during which time blood was taken from the cannulated animals (one or two animals were cannulated). If no animals were cannulated, one or two animals were removed at the time of incapacitation or at the end of the exposure to obtain blood samples.

The biological endpoints examined were incapacitation and lethality. Incapacitation was measured by the hind-leg flexion conditioned avoidance response test developed by Packham et al. [6]. The time-to-incapacitation of each of the six animals in each experiment was noted and the mean and standard deviation of those times were calculated for each mass loading. In addition, the EC_{50} [mass loading of material per unit chamber volume (mg/l) that caused 50 percent of the animals to become incapacitated in the 30 minute exposure] was determined. The lethality endpoint required by the proposed test method was the LC_{50} for 30 minutes plus 14 days [the mass loading of material per unit chamber volume (mg/l) which was necessary to cause 50 percent of the animals to die during the 30 minute exposure plus a 14 day post-exposure observation period]. In addition, the LC_{50} for the 30 minute exposure period was also calculated when enough data were available. Both the EC_{50} 's and LC_{50} 's, their 95 percent confidence limits, and the slopes of the concentration-response curves were calculated via the statistical method of Litchfield and Wilcoxon [7]. (Concentration, when indicating the total amount of combustion products generated during an experiment, is defined as the mass loading of material per unit volume of exposure chamber.)

3.0 THE INTERLABORATORY EVALUATION OF THE 1980 TEST METHOD

The purpose of the ILE was to determine the reproducibility of the test results from different laboratories. In addition, the ILE helped refine the necessary conditions of the test. Nine laboratories initially agreed to follow the 1980 test procedure and to determine or to monitor the following parameters: the autoignition temperature of each required material; the temperatures generated in the cup furnace and in the animal exposure chamber; the CO, CO₂, and O₂ concentrations generated during each material's decomposition in the flaming and non-flaming modes and, if conditions warranted, at 440°C; time-to-incapacitation of each animal exposed and the mean and standard deviation of the time-to-incapacitation for all six animals used at each mass loading; the number of animals incapacitated or killed during the exposure at each mass loading and the number who died during the 14 day post-exposure observation period; and the COHb at incapacitation and at the end of each 30 minute experiment. Enough experiments were to be performed to allow an EC₅₀ for the 30 minute exposure and an LC₅₀ for the 30 minute exposure plus the 14 day post-exposure observation period to be statistically calculated by NBS from the data. The paper by Litchfield and Wilcoxon [7] describes the procedure for calculating the LC₅₀ values. (Formulas from the appendix of the Litchfield and Wilcoxon paper were used instead of the nomographs included in the text.)

The seven participants in the ILE are listed alphabetically in table 1. Each laboratory was assigned an identification number which will be used throughout this report and in tables of results. These numbers do not correspond to the order of the laboratories given in table 1. Twelve materials, both synthetic and natural and having a broad range of thermal and toxicological properties, were chosen for this study. The list of materials and the abbreviations used throughout this paper are shown in table 2. A common stock of materials was collected, stored, and sent to the participants by NBS. The detailed composition of each of the polymers and co-polymers was not available. Assemblages of non-uniform structure, such as carpets or layered wall materials, were not tested in this ILE.

The statistical design of the ILE was generated by one of the participating laboratories by randomly numbering each material (1 to 12) and each laboratory (1 to 9) and then selecting three materials which were assigned at random to a laboratory for study. In addition, each laboratory experimental design is shown in table 3. When two laboratories (7 and 9) withdrew, the design was changed. Some of the other laboratories agreed to study additional materials and NBS agreed to examine all twelve. These changes are noted in table 3 by the letter A representing additional materials, and N.D. representing not done. The ultimate result was that laboratories 1 and 8 tested four materials, laboratories 3 and 5 tested five materials, laboratory 2 evaluated six materials, laboratory 4 studied seven and laboratory 6 (NBS) evaluated all twelve. Thus all materials except poly(vinyl chloride) (PVC) were examined by at least three laboratories; PVC was studied by only two.

All participants were required to complete standardized data sheets for each material in each temperature mode (tables 4a,4b,4c). All data sheets were sent to NBS for analysis.

4.0 VARIATION IN EQUIPMENT AND TECHNIQUES

Although all participants were requested to follow the 1980 test method as outlined in section 2.0, certain options were left to the discretion of the investigators, for example, the type of analytical equipment, the rat strain, the animal restrainer material, and the method of blood sampling. Other items, although specified, were still varied by some of the laboratories. These differences are summarized in tables 5 and 6 and will be discussed in the following sections.

4.1 Analytical Instrumentation

The 1980 test method required the continuous or intermittent (every 5 minutes) measurement of CO, CO₂, and O₂. For CO and CO₂, five laboratories (1, 2, 4, 5, and 6) used continuous flow nondispersive infrared analyzers which monitor the absorption of specific wavelengths characteristic of the chemical species of interest (table 5). Two laboratories (3 and 8) made intermittent measurements using gas chromatographs equipped with thermal

conductivity detectors. Oxygen measurements were performed with paramagnetic (laboratory 1), polarographic (laboratories 2, 5, and 8), galvanic (laboratories 4 and 6), and chromatographic (laboratory 3) techniques.

Although the test method did not require the determination of hydrogen cyanide (HCN), three laboratories (1, 3, and 6) volunteered to measure HCN in the combustion atmospheres of those ILE materials containing nitrogen. Laboratories 3 and 6 analyzed HCN with a gas chromatograph equipped with a thermionic detector [8]. Laboratory 1 used a specific ion electrode.

4.2 Furnace

All laboratories were to use a cup style furnace based on the design of Potts and Lederer [4]. In this system, a quartz beaker with a thermocouple well was surrounded by ceramic with recessed heating elements, all of which was encased in a galvanized steel box (fig. 2). The quartz beaker in which the sample was degraded was heated to a predetermined temperature which was monitored by a temperature controller. The materials were degraded by a combination of convective, conductive, and radiant heat.

In the ILE, the furnaces were made by the participants and the sizes of both the furnaces and quartz cups differed to some degree (table 6). Most of the laboratories used a cup of approximately 300 ml volume; however, lab 6 (NBS) also tested a cup furnace with a capacity three times greater (954 ml) and laboratory 8's cup was much smaller than the others with a capacity of only 79 ml. The two cup sizes used by NBS are designated 6a (362 ml) and 6b (954 ml) in the tables of results.

4.3 Exposure Chamber

The exposure chambers of all the laboratories except laboratory 8 were based on the design shown in figure 1 and had a volume of approximately 200 liters (table 6). The exposure chamber of laboratory 8 was separated from the furnace and is shown in figure 4. This figure also shows rotating cages which were present but not used in this ILE.

4.4 Animal Information

The information pertaining to the animals is summarized in table 6. All laboratories exposed male rats to the combustion atmospheres in a head-only mode, not whole body. The strain of rat varied; four laboratories used Sprague-Dawley, three laboratories used Fischer 344, and one laboratory used Long-Evans. The animals upon receipt from the supplier were between 2 and 4 months of age and were allowed to acclimate to laboratory conditions for 7-14 days before testing. All laboratories except 8 exposed six animals per test (at each mass loading); laboratory 8 exposed only three, but repeated each exposure four times--twice for lethality measurements and twice for incapacitation measurements.

Laboratory 3 cannulated three animals and laboratory 6 cannulated two animals per test in order to sample blood during the exposure without removing the animals from the combustion atmosphere. Laboratory 5 cannulated one or two animals occasionally. Laboratory 3 kept the cannulated animals throughout the 14 day post-exposure observation period, but laboratory 6 sacrificed the cannulated animals after the 30 minute exposure and only kept the non-cannulated animals for post-exposure observation. The other laboratories (1, 2, 4, and 8) removed their animals from the chamber and obtained blood via cardiac puncture (with and without open chest) or intraorbital venous puncture. A co-oximeter was used by all laboratories to analyze the percent carboxyhemoglobin present in the blood.

For testing, animals were placed in aluminum (laboratories 2, 5, 6) or plastic restrainers (laboratories 1, 3, 4, 8) which were inserted into the port-holes along the front of the chamber (fig. 1) such that only the animals' heads were exposed to the combustion atmosphere. The animals were observed for both time-to-incapacitation and death. Incapacitation was measured by the hind-leg flexion conditioned avoidance behavioral model [6] in which wires are attached to one of the hind legs of the rat such that the animal receives an electrical shock whenever the foot touches a metal plate located below the animal. The shock current varied among the laboratories from 1-3 ma to about 13 ma (table 6). The animals learned rapidly (approximately 15 min) prior to the test not to touch the plate and were considered incapacitated when they

failed to respond to the shock. Laboratory 3's electrical circuitry was designed such that after an animal became incapacitated and the electricity to that animal was shut off, the electrical current to the rest of the animals increased.

5.0 RESULTS AND DISCUSSION

5.1 Temperature Measurements

5.1.1 Autoignition Temperatures

Each laboratory was responsible for independently determining the autoignition temperature of each of their designated materials plus Douglas fir. The temperatures found by the laboratories are summarized in table 7 and illustrated in figures 5a and 5b. The solid black part at the top of the bars in these figures indicates the intralaboratory variability (low and high values) in the autoignition temperature data. Part of this variability was attributed to the initial determination of the autoignition temperature with relatively small sample sizes. In the course of the non-flaming experiments (25°C below the autoignition temperature), larger sample sizes sometimes underwent an exothermic reaction which caused the sample to ignite. Consequently, both the autoignition temperature and the non-flaming temperature were lowered. Laboratory 4 did not submit autoignition temperatures for most of their materials and the missing values were estimated by calculating the mean values between the highest non-flaming temperature and the lowest flaming temperature reported (table 7 and figures 5a and b).

Good agreement among the laboratories is seen by the small scatter of data around the mean of the autoignition temperature for each material (table 7). Most of the standard deviations ranged between 12 and 35°C; only polystyrene had a standard deviation which exceeded these values.

5.1.2 Flaming and Non-flaming Temperatures

Once the autoignition temperature of a material was determined, experiments were to be conducted 25°C above (flaming mode) and 25°C below this temperature (non-flaming mode). If this requirement were followed rigorously,

there would be no need to report the flaming and non-flaming temperatures. This information is provided, however, because some of the laboratories did not perform their experiments on any one material at a constant temperature and, in some cases, they did not adhere to the 25°C requirement. The flaming temperatures are listed in table 7 and figures 6a and 6b. The non-flaming temperatures are in table 7 and figures 7a and 7b. The black part of each bar in these figures indicates the range of temperatures that a laboratory used in the examination of any one material (their intralaboratory variability).

The variation in methodology between laboratories was used to evaluate the sensitivity of the results to furnace temperature. For example, the autoignition temperature of acrylonitrile butadiene styrene (ABS) was determined to be 555°C by laboratory 3 and to be 500°C by laboratory 5. Both of these laboratories then used flaming temperatures of between 694°C and 725°C disregarding the specification of the test method to do flaming experiments 25°C above the autoignition temperature. However, the LC₅₀ results from these laboratories for flaming ABS are not significantly different from the other two laboratories who did follow the specification. On the other hand, data from laboratory 4 showed that in the non-flaming mode the LC₅₀ values for modacrylic decreased as the furnace temperature approached the autoignition temperature, indicating the non-flaming mode results may be more sensitive to furnace temperature (table 8).

5.1.3 Chamber Temperatures

To minimize the effects of heat stress on the animals, the 1980 test method recommended that the temperature of the exposure box not exceed 35°C at any time during the 30 minute exposure period. Temperatures were measured close to the animals' noses and also in the middle of the exposure chamber. Both average and maximum temperatures were reported for the 30 minute period for each experiment.

Preliminary analysis of the data indicated that the temperatures measured in the vicinity of the animals' noses were usually lower than those measured in the middle of the exposure chamber. Therefore, to estimate the heat stress that the animals experienced at any time, only the temperature readings near

the animals' noses were considered. The highest nose temperature during the 30 minute exposure period for each material over the mass loading range studied was reported to NBS and is listed in table 9.

These data indicated that a maximum nose temperature of 35°C as specified in the proposed test method was exceeded for many materials. In the flaming mode, examination of the maximum nose temperatures showed results above this specified limit in 23 out of 29 sets of experiments. In the non-flaming mode, the maximum temperature rose above 35°C in 13 out of 27 sets of experiments. The worst case, a maximum temperature of 109°C, was observed by laboratory 6 (NBS) during the decomposition of 41 mg/l of wool in the flaming mode. In this experiment, 50 percent of the exposed animals died. Another wool experiment at a lower mass loading (22 mg/l) had a very similar temperature profile, but no animals died (fig. 8). These results indicated that temperatures of this magnitude for short durations do not necessarily produce enough heat stress to cause death of animals exposed in a head-only mode.

Instead of the maximum temperature, the average temperature over the exposure time may be a better indicator of the total heat stress experienced by the animal. Table 10 shows the highest average nose temperature found during the flaming and non-flaming decomposition of each material by each laboratory. It was on the basis of these data that the specified upper limit of 35°C at any time during the exposure was changed to an average temperature limit of 40°C. Only three values out of 77 exceeded this limit and one of these values is suspect as the non-flaming average nose temperature was greater than the average flaming temperature, a situation unlikely to occur.

It is not clear from this study what temperature level or exposure time is necessary before the head-only exposed animals will show visual or physiological effects of heat stress. It is also not known what temperature level is needed to produce additive or synergistic effects with the toxicants that also are likely to be present.

5.2 Chemical Measurements

The 1980 version of the test method [2] required the continuous or intermittent measurement of CO, CO₂, and O₂ in the exposure chamber atmosphere during the thermal degradation of materials. In addition, HCN was measured by some laboratories if a nitrogen-containing material was tested. In this study, the average concentration of a chemical species for any one mass loading of material (one experiment) was obtained by integrating the area under the instrument response curve and dividing by the duration of the experiment, 30 minutes.

5.2.1 Carbon Monoxide

CO concentrations generated by the thermal decomposition of each material except polytetrafluoroethylene (PTFE) were measured. The CO in the combustion products from PTFE was not routinely measured in order to avoid possible instrument damage by fluorides. Laboratories 1 and 4, however, measured the concentration of CO in a few PTFE experiments and found that the average concentration over the 30 minute exposure was never greater than 60 ppm.

To compare the results from various laboratories, the average concentration of CO generated over the 30 minute experiments was plotted against the mass loading of material per unit chamber volume (mg/l). The slopes of the curve for each laboratory were calculated by a least squares linear regression analysis.

Douglas fir results are shown in figure 9A (non-flaming mode) and figure 9B (flaming mode), in which the dashed lines indicate the laboratories with the greatest and smallest slopes, and the solid line is the slope of results from all the laboratories. The production of CO in the non-flaming mode was proportional to the mass of material loaded into the cup furnace up to 30 mg/l. When the sample loading was greater than 30 mg/l (for the 200 liter chamber, this is a 6 gram sample), the increase in CO with increasing mass was no longer linear. This lack of linearity was also noted for flaming Douglas fir when the mass loading per the chamber volume exceeded 50 mg/l (fig. 9B). This occurrence was attributed to the inability of the cup

furnace to decompose the large mass of material in the allotted time. (Note, however, that this overload was not visually apparent as this mass of material fits easily into the cup.) An increase in the size of the cup furnace to 954 ml did not correct this problem. Concentrations of CO from mass loadings of Douglas fir above 30 mg/l (non-flaming) and 50 mg/l (flaming) were not included in the determination of slopes via least square linear regression analysis or in the statistical analysis as presented below. No such overloading of the cup was observed for the other materials examined during this study.

Within the limits of the cup heating capability as noted above, the CO production is proportional to the mass loading/chamber volume. Consequently, the statistical analysis of the CO data across laboratories was performed on CO concentrations averaged over the 30 minute exposure and normalized to the mass of material loaded into the cup furnace divided by the chamber volume:

$$\frac{\text{average CO over 30 minutes}}{\text{mass loading/chamber volume}} = \frac{\text{ppm}}{\text{mg/l}}$$

This normalization enabled the comparison of data from different laboratories even when materials were tested at different mass loadings. Each laboratory's mean values and standard deviations of the ratios of average CO (ppm) to mass loading/chamber volume (mg/l) for each material except Douglas fir are shown in table 11. Douglas fir results are presented along with the data used to calculate the means and standard deviations in tables 12 and 13.

To facilitate comparisons of results between a pair of laboratories chosen from the participating laboratories, an interval "w" was computed such that any two laboratory results may be considered (1) similar if the absolute difference between means is less than "w" or (2) not similar if the absolute difference between means exceeds "w". The "w" statistic was computed as shown in table 14 following the procedure in section 3.4 of reference [9], and uses the within-laboratory precision as a measure to compare differences of means among laboratories.²

As all the laboratories examined Douglas fir, the "w" values for the Douglas fir CO results were computed and those laboratories with similar and

dissimilar results were determined (table 14). More similarity was seen in the non-flaming mode in which only the results from laboratory 8 were different from laboratories 2, 3, 5, 6a and 6b. All the other laboratories showed no significant differences in their CO results. Although laboratory 8 was similar to laboratories 1 and 4, the reason that its values were significantly different from most of the other laboratories is likely related to its very different exposure chamber and combustion furnace (fig. 4 and table 6). The results of the flaming mode tests showed that most of the laboratories were statistically similar with at least three other laboratories. Laboratory 3, however, was statistically different from all the other laboratories. The reason for this difference is not clear, however, it should be noted that the better intralaboratory precision for the flaming mode resulted in a smaller "w" value and thus a more stringent interlaboratory comparison which indicated more dissimilarities.

One of the factors which could influence the material decomposition and CO generation is the size of the cup furnace. As noted in table 6, most laboratories had cup volumes of approximately 300 ml (236 - 362 ml). Laboratory 8 used a 79 ml cup and laboratory 6 (NBS) used a 954 ml cup in addition to their 362 ml cup. Figure 10 shows the NBS results on CO generation from flaming Douglas fir in both the small and large cup furnaces. When the average CO generated over the 30 minute experiments was plotted against the mass loading/chamber volume, the larger furnace results had a slope that was 12 percent steeper than the smaller furnace results irrespective of whether the material was degraded in the flaming mode or non-flaming mode. As the within-laboratory variation in CO generation from Douglas fir [as indicated by the relative standard deviations (tables 12 and 13)] ranged from 6-26% in the flaming mode and from 6-34% in the non-flaming mode, a 12 percent difference in slope due to the cup furnace size is probably not significant. Since the larger cup can accommodate larger samples of low density materials, the recommended size of the cup furnace was increased from 300 ml to 1000 ml.

²We note that a number of assumptions underlying this comparison procedure may not be fulfilled, namely: the presence of outliers, unequal number of test results, and unequal variances. Dunnett [10, 11] treats unequal sample sizes and unequal variances, but the presence of outliers still poses a different problem.

5.2.2 Carbon Dioxide

CO₂ is produced during the combustion of all organic materials, and the ratio of CO/CO₂ is one indicator of the completeness of combustion. Toxicologically, CO₂ is not lethal until the concentrations reach 12 percent (120000 ppm) [12], levels far greater than those observed during this study. The maximum average levels of CO₂ generated over 30 minutes from each material observed by a laboratory and the respective mass loadings are shown in table 15. However, CO₂ does act at lower concentrations (1.8 percent or 18000 ppm) to stimulate the respiratory center of the brain causing increased respiratory rates and thus acts to increase the rate at which other toxic gases are inhaled. It is important to note that the measured concentrations of CO₂ are a result of both material combustion and animal respiration in addition to the CO₂ content already present in ambient air. In experiments at NBS, the normal respiration of six rats placed in the closed exposure chamber for 30 minutes (with no combustion) produced an average concentration of 2900 ppm of CO₂ and a maximum concentration of 6900 ppm. These results, however, could not be used as control values to determine the CO₂ from only the material decomposition as the animals' respiratory rates were also dependent upon which material was burned and the other toxicants present.

For the above reasons, statistical analyses were not performed on the CO₂ data. The average values of CO₂ were calculated over the 30 minute exposure time and normalized to the mass loading per unit chamber volume. The mean and standard deviations of all the normalized values for a material in a particular mode (flaming or non-flaming) found by each laboratory are tabulated in table 16.

5.2.3 Oxygen

Both the material combustion and the animal respiration rate will reduce the oxygen levels in the animal exposure chamber. To prevent the compounding of the toxicity information with possible O₂ deprivation effects, the 1980 method specified that the O₂ concentration not fall below 18 percent. The average percent O₂ over the 30 minute exposures and the minimum percent O₂ were determined by the ILE participants and reported to NBS. Preliminary

analysis of the data showed that the average O_2 concentration was a better indicator of O_2 deficiency than the lowest O_2 reading. In other words, an individual reading does not accurately represent the concentration that the animal experiences over the 30 minute exposure.

The minimum average O_2 concentrations from each laboratory for each of their respective materials are summarized in table 17. In the non-flaming mode and at $440^{\circ}C$, only two sets of data out of 58 fell below 18 percent and those two were 17.5 percent. In the flaming mode, on the other hand, 17 sets of data out of 39 were below 18 percent. However, only one value (14.2 percent for flexible polyurethane) was below 16 percent. Recent studies by Matijak-Schaper and Alarie [13] indicated that O_2 levels could fall as low as 10 percent before the respiratory rate of their experimental animals (mice) showed a slight decrease; therefore, the requirement of maintaining the O_2 level at no lower than 18 percent in the exposure chamber was changed in the 1982 test method [3] to an average concentration of no lower than 16 percent.

5.2.4 Hydrogen Cyanide

The determination of HCN concentrations was not required by the 1980 method. However, three laboratories (1, 3, and 6) did measure the HCN concentrations generated by the thermal decomposition of the nitrogen-containing materials [ABS, flexible polyurethane, modacrylic, poly(vinyl chloride) with zinc ferrocyanide, rigid polyurethane, and wool]. Not all of the three laboratories, however, measured HCN from all six materials in all three modes (flaming, non-flaming and $440^{\circ}C$). Table 18 shows the average concentrations of HCN at each mass loading.

The generation of HCN was found to be proportional to the mass loading per unit chamber volume (mg/l). The results for non-flaming ABS are shown in figure 11. To compare the other material results from the three laboratories, the HCN concentrations were normalized to the mass loadings/chamber volume (table 19). Although a statistical analysis of the results was not performed due to the limited amount of data, visual inspection shows reproducibility to within a factor of 2.5 as measured by the ratio of the maximum value to the minimum value.

5.3 Animal Measurements

5.3.1 Incapacitation

The biological endpoints examined in this ILE were incapacitation of the animals during the 30 minute exposure and death during the 30 minute exposure and 14 day post-exposure observation period. The experimental results on incapacitation provided by the ILE participants were analyzed on the basis of both time-to-incapacitation and the numbers of animals incapacitated at each mass loading in order to determine an EC_{50} [mass loading/chamber volume (mg/l) necessary to cause 50 percent of the animals to become incapacitated in the 30 minute exposure]. All ILE participants used the hind-leg flexion conditioned avoidance response as the behavioral model for incapacitation [6].

Time-to-incapacitation can be examined in two ways. In the first, the mass loading of material is kept constant and the times-to-incapacitation of each of the six exposed animals are measured during each experiment which lasts until all animals are incapacitated. In this manner, the mean time-to-incapacitation could be compared for the same mass loading of materials. This procedure worked better for some materials than for others. Those materials whose combustion products caused post-exposure deaths rather than within-exposure deaths produced extremely variable results when examined by this method. Poly(vinyl chloride) is an example of such a material. Upon thermal decomposition, PVC produces HCl , a potent toxicant and highly irritating acid gas. The irritating effects of the combustion products from this material are so intense that the animals temporarily fail to react to the shock. This seemingly incapacitated state will recur repeatedly during the exposure. Within the same experiment the actual time of incapacitation varied widely (table 20) and some animals died before others were incapacitated. Other materials, mainly those which produced within exposure effects, showed good repeatable results with this method.

In the second procedure, the exposure duration is set (for example, at 30 minutes) and the mean and standard deviation of the time-to-incapacitation for the exposed animals is calculated for different mass loadings of material. The lower the mass loading, the more time needed to incapacitate the

animals. These points were represented by a function which asymptotically approaches a threshold time-to-incapacitation on one axis and a concentration of material needed to produce incapacitation in the specified time limit on the other axis.

The mean times-to-incapacitation that were obtained in each mode were used to best fit a hyperbolic curve ($Y = Q + R/X$). Six examples of these hyperbolic curves for Douglas fir in the non-flaming mode are shown in figure 12. The hyperbolic equations are shown on each graph. Similar time-concentration hyperbolas were generated for all the materials in both flaming and non-flaming modes from the NBS data and that supplied to NBS by the other laboratories. The Q and R coefficients calculated for each material in each mode from all the laboratories are presented in table 21. Visual inspection and comparison of the coefficients indicate the differences between the curves. The problems that arise with the use of the hyperbolic curves for the analysis of relative toxicity of the combustion products of materials are: (1) differences in time-to-incapacitation of 5-10 minutes are equivalent to 16-30% of the total 30 minute time frame, i.e., substantial experimental scatter is inevitable, (2) the 30 minute exposure time limits the number of data points at lower mass loadings (where less than six animals are incapacitated), and (3) comparison of the various curves to determine relative toxicity of materials is difficult.

Another means of analyzing the incapacitation data is to determine the EC_{50} . The percent of animals incapacitated at each mass loading tested is plotted on logarithmic probability paper to obtain a concentration-response curve. The EC_{50} 's and their 95% confidence limits were statistically determined by NBS for all the materials in both the flaming and non-flaming modes by the method of Litchfield and Wilcoxon [7]. In some cases, however, a small change in concentration (e.g., 0.5 mg/l) would cause the number of animals incapacitated to change from 0% to 100%. In these cases, the EC_{50} was obtained graphically from a plot of the data in which successive data points were connected by straight line segments (fig. 13). Whenever a value was estimated by this latter method, the approximate sign (\sim) is placed before that value and the extremes used to obtain that value are placed in brackets. Table 22 shows the EC_{50} values (those statistically determined and those approximated) with their 95% confidence limits in parenthesis.

As a result of the analysis of the data provided by the ILE participants, incapacitation (as evaluated on the basis of the hind-leg flexion behavioral model) was found to provide less toxicological information (did not detect materials which caused post-exposure effects), was a less sensitive indicator of acute toxicity (false positives were noted with smoke containing high concentrations of irritants) and was a more difficult procedure to measure practically than the biological endpoint of lethality. Additional incapacitation results and a detailed explanation for excluding the incapacitation endpoint from the 1982 test method are provided in reference [3].

5.3.2 Lethality

The assessment of the acute inhalation toxicity of combustion products in the 1980 version of the test method is based on the determination of a statistically calculated LC_{50} [the mass loading of material/chamber volume (mg/l) necessary to cause 50 percent of the test animals to die within the 30 minute exposure and/or 14 day post-exposure observation period]. Briefly, the determination of an LC_{50} is as follows: Six animals are exposed to the combustion products from a specific mass loading of material and the percent of animals which respond in the set time period are noted. A new set of animals are then exposed to a higher or lower mass loading in order to generate a concentration-response curve in which the percent deaths are plotted as a function of the amount of material loaded into the furnace (fig. 13). This concentration-response curve provides a means of estimating the LC_{50} . The actual statistical determinations of the LC_{50} 's and the slopes of the concentration-response curves from the data provided by the ILE laboratories were carried out at NBS according to the method of Litchfield and Wilcoxon [7]. Table 23 shows the LC_{50} values and their 95 percent confidence limits calculated for the 30 minute exposure and 14 day post-exposure observation period from the data provided by all the laboratories. Table 24 shows the concentration-response slopes for the same data. The 95% confidence limits shown in tables 23 and 24 are indicative of the intralaboratory variability. Visual inspection of the LC_{50} values listed in table 23 shows the reproducibility between laboratories for each material in each mode is within a factor of two except for PTFE and PPS in both the flaming and non-flaming modes and wool in the flaming mode (table 25).

No laboratory found the LC₅₀ (30 minutes and 14 day) values determined at the 440°C furnace temperature to be lower than the lowest LC₅₀ value determined in either the flaming or non-flaming mode. As the test method is designed to evaluate materials under their most toxic conditions, the 440°C temperature mode was made optional in the 1982 version of the test method [3].

The sensitivity of the LC₅₀ values to the cup furnace size was examined. Complete concentration-response curves were generated by NBS, and separate LC₅₀ (30 minutes and 14 day) values were calculated for Douglas fir decomposed in the NBS large furnace (954 ml) and the NBS small furnace (362 ml). The flaming and non-flaming results are shown in table 23 under the material heading Douglas fir and laboratory numbers 6a (small furnace) and 6b (large furnace). Visual inspection of these LC₅₀ values and their 95% confidence limits indicates that these values were not sensitive to the different size cup furnaces tested by NBS.

5.3.3 Blood Measurements

Each laboratory participating in the ILE measured both carbon monoxide levels in the exposure chamber atmosphere and blood carboxyhemoglobin (COHb) in each test. If the animals were cannulated as described earlier (section 2.0), arterial blood could be sampled during the exposure without removing the rats from the exposure chamber. If the rats were not cannulated, they were removed from the chamber before sampling. Those laboratories who did or did not routinely use cannulated animals are identified in section 4.4.

A great amount of blood data was generated during this study. Table 26 shows the blood parameters measured by NBS and the mean and standard deviations determined in 138 - 180 control animals. This table provides an indication of the intralaboratory precision of one of the laboratories, namely NBS, in the determination of these values. (A more extensive evaluation of the blood measurements will be published by NBS in a separate report.) For the purposes of this report, the blood values of interest were the COHb values determined at the biological endpoints of incapacitation and death. There were two reasons for this interest: (1) to determine if the values were reproducible across laboratories and (2) to examine the COHb levels in order

to assess whether carbon monoxide was the principal toxicant responsible for the death of the animals. This latter reason is the primary purpose for including the COHb measurements in the test method.

Table 27 shows the COHb levels and corresponding average concentrations of CO calculated for each material examined by each laboratory at two biological endpoints: (1) the LC₅₀ (30 minutes), and (2) the EC₅₀ (30 minutes). The LC₅₀ for 30 minutes is used here since all the blood samples were taken at the end of the 30 minute exposure and the COHb values are related to the average CO concentration to which the animals are exposed over the 30 minutes. In addition, the COHb levels determined at the time of incapacitation are also provided in table 27. To determine the percent COHb at the LC₅₀ and EC₅₀, the COHb values that were obtained at the end of each exposure were plotted against the mass loading of material/chamber volume (mg/l). The LC₅₀ and EC₅₀ values were superimposed upon the graph and the percent COHb at that mass loading was determined (fig. 14). To determine the percent COHb at the time of incapacitation, the mean and standard deviation of the percent COHb at the time of incapacitation for each material was calculated utilizing all values of COHb obtained from one laboratory at incapacitation regardless of mass loading. The mass loading would change the actual time of incapacitation, but the percent COHb at incapacitation should theoretically remain constant. The difference between the percent COHb at the EC₅₀ and the percent COHb at the time of incapacitation is that the former is calculated from blood taken at the end of the exposure at the mass loading which causes 50% of the animals to be incapacitated. The latter is the mean of the actual percent COHb calculated from blood taken from animals as soon as possible after the animal became incapacitated, although there is always a time lag during which the COHb levels will increase if the animal is not removed from the exposure atmosphere or decrease if the animals are removed.

A distribution-free statistical test (the sign test) was performed on the COHb values for all the laboratories to determine if any laboratory's results were consistently lower or higher than the overall mean results[14]. In table 28 (LC₅₀, 30 minutes + 14 days), table 29 (LC₅₀, 30 minutes), table 30 (EC₅₀), and table 31 (time-to-incapacitation), the mean COHb value for all the laboratories for a particular material in one mode was subtracted from the value

obtained by each laboratory. When the value from an individual laboratory was lower than the mean, the difference is listed as a negative value and when the laboratory value was higher than the mean, the difference is listed as a positive value. One can now check how many times a laboratory had negative versus positive numbers and how many times a laboratory had the lowest value or the highest value. The results of this statistical sign test on COHb at the LC₅₀ (30 minutes + 14 days) and LC₅₀ (30 minutes) are shown on the bottom of tables 28 and 29, respectively. These tables show that laboratories 1 and 4 were frequently lower than the mean; whereas laboratories 3 and 6 were frequently higher than the mean. (In these two tables, only laboratory 6b's results are statistically significant at the 5% level. However, in many cases, the split between the number of negative effects and positive effects was not sufficient to evaluate for significance, i.e., at least a 0/6 split was necessary.)

These results reflect the method of obtaining blood. Those laboratories that took blood via cannulated animals without removing the animals from the smoke atmosphere had values that were higher than the mean. Those laboratories that never cannulated but rather removed their animals from the exposure chamber to obtain blood had COHb values that were lower than the mean. Post-exposure recovery rates from pure CO exposures in rats is very rapid (NBS data, to be published). At these high COHb levels, the COHb drops 50% in 15 minutes, which indicates that a short delay in obtaining the blood samples after removal of the animal from the exposure chamber will produce low values.

A similar evaluation of the incapacitation data (bottom of tables 30 and 31) does not show a relationship with the method of obtaining blood. In this case, there is both variability in time-to-incapacitation and variability in time-to-blood sampling. For example, reference to the EC₅₀ values in table 22 shows that laboratory 3 consistently obtained lower EC₅₀ values. Thus it is not surprising that laboratory 3 has a high percentage of low COHb values in both tables 30 and 31.

The second reason for examining the blood COHb data is to determine whether CO was the primary toxicant generated by the thermal degradation of

the material being tested. Pure gas studies at NBS have shown that an average concentration of 5000 ppm of CO and 89% COHb are necessary to kill 50 percent of the rats in 30 minutes under the laboratory conditions specified by this toxicity test method. Table 27 shows that 5000 ppm of CO and 89% COHb are higher values than those determined for any of the twelve ILE materials at the mass loadings/chamber volume needed to kill 50 percent of the rats within the exposure time (30 minutes). The lower levels of CO and COHb found at the LC₅₀ (30 minutes) for the various materials indicates that other factors or toxicants are acting in conjunction with the CO to produce the within-exposure deaths. As the pure CO experiments did not cause post-exposure deaths, the death of animals during the 14 day post-exposure observation period must be due to other toxicants or unknown factors.

6.0 SUMMARY

Seven laboratories from academia, industry, and government participated in an interlaboratory evaluation of the 1980 version of the toxicity test method developed by the National Bureau of Standards. This interlaboratory evaluation was designed to serve two purposes. The first was to examine the reproducibility of results from the toxicity test method across laboratories. The second purpose was to test the operability of the procedure and to identify the modifications necessary to improve the method. Although the ILE was not designed to specifically examine the repeatability of experimental results within each laboratory or the sensitivity of the test method results to variations in procedures and equipment, these factors are examined in this report wherever possible.

This ILE was not a round robin, i.e., not all laboratories examined all materials or used exactly the same methodology and instrumentation. In addition, the test method itself continuously changed during this study, primarily as a result of the information collected and presented to the ad hoc working group by the ILE participants. The small number of laboratories which examined some of the materials and the variations in procedure between laboratories (e.g., concentrations tested) limited the use of some kinds of statistical analyses on the reproducibility of results.

Data collected during this study and examined for reproducibility of results across laboratories included: a) the autoignition temperatures, b) the CO concentrations, c) the HCN concentrations from the decomposition of nitrogen-containing materials, d) the LC₅₀ values calculated for the 30 minute exposures and 14 day post-exposure observation periods for each material, and e) the COHb values. The main conclusions follow:

1. The autoignition temperatures independently determined for each material showed good agreement across laboratories.
2. CO concentrations generated by the decomposition of Douglas fir were more reproducible across laboratories in the non-flaming mode than the flaming mode. However, even in the flaming mode, many of the laboratories were statistically similar.
3. HCN results across laboratories showed reproducibility to within a factor of 2.5.
4. A change in the cup furnace volume from 300 ml to 1000 ml does not significantly influence the CO concentrations generated from the decomposition of Douglas fir nor the subsequently determined LC₅₀ values. Also, this increase in the volume of the cup furnace did not solve the problem of overloading the heating capability of this particular furnace design. An increase in cup furnace volume was recommended, however, in order to accommodate greater quantities of low density materials.
5. The biological endpoint - the LC₅₀ values for the 30 minute exposure and 14 day post-exposure observation period - appears to be less sensitive to changes in the flaming decomposition temperature than to the non-flaming decomposition temperature.
6. Brief excursions of the chamber temperatures to as high as 100°C did not produce deaths.
7. The LC₅₀ (30 minutes and 14 day) values were reproducible to within a factor of two for all materials decomposed in both the flaming and non-flaming modes except for polytetrafluoroethylene, polyphenylsulfone and wool.

8. The COHb data indicated that the results were very dependent upon the time of sampling and whether or not the animals were removed from the toxic atmospheres to obtain samples.

The second purpose of the ILE - to determine the modifications necessary to improve the effectiveness of the test method - was fulfilled by the refinements which resulted in the 1982 NBS test method [3]. The changes which resulted from the ILE are presented here:

- examination of materials at 440°C is now optional,
- the recommended size of the cup furnace has been increased from 300 ml to 1000 ml,
- the highest average chamber temperature permitted in the vicinity of the noses of the animals for the 30 minute exposure was raised to 40°C,
- the minimum average oxygen level permitted in the chamber was lowered from 18 to 16 percent,
- for the 30 minute exposure, the only biological endpoint now required is an LC₅₀ [the mass loading/chamber volume (mg/l) which causes lethality in 50 percent of the animals in the 30 minute exposure plus a 14 day post-exposure observation period]. The incapacitation endpoint has been eliminated, and
- blood from non-cannulated animals must be taken in the first 5 minutes after the end of the exposure.

Other changes in methodology which resulted from information presented at the ad hoc working group meetings are detailed in the report on the 1982 version of the test method [3].

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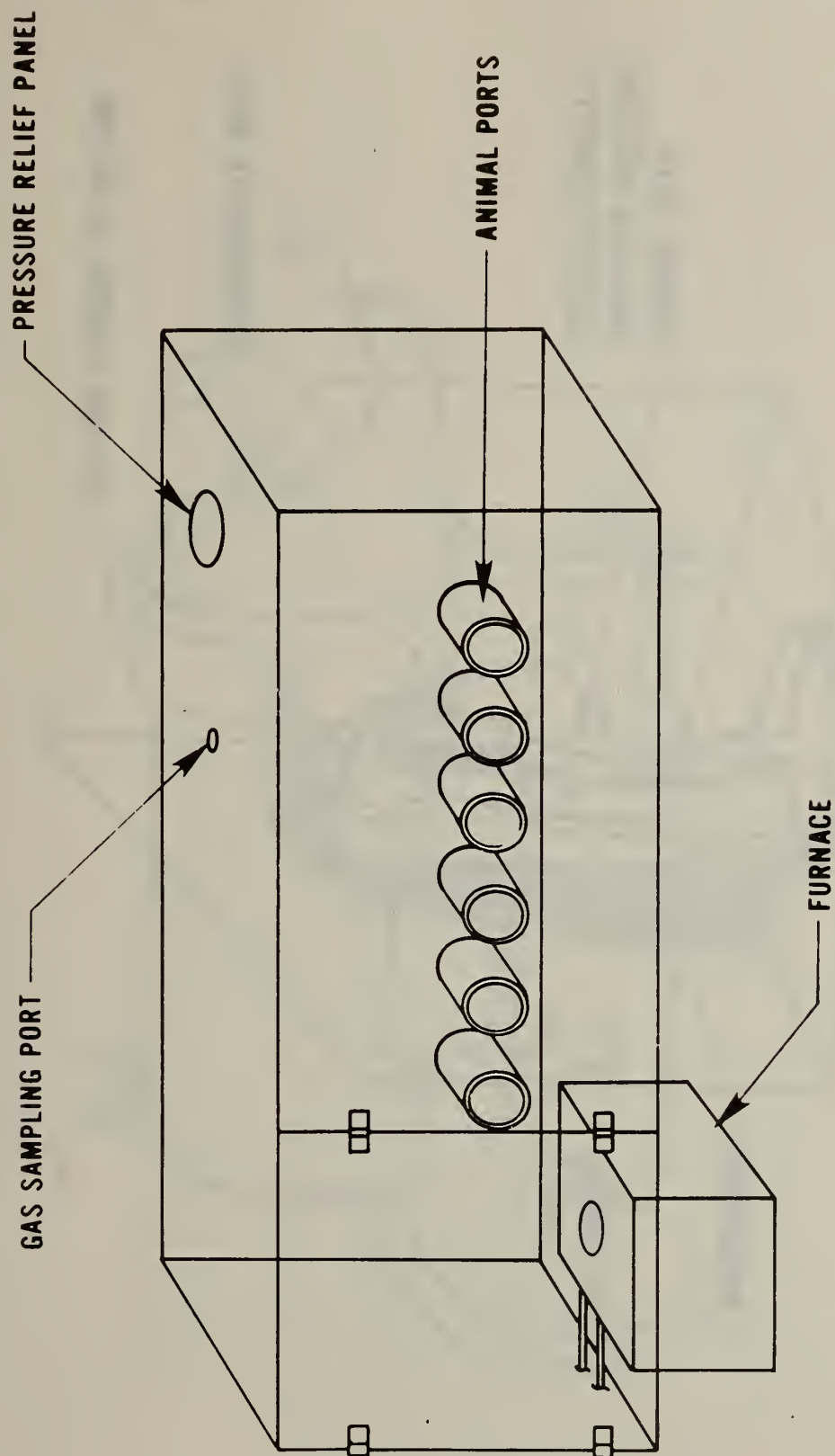


Figure 1. Exposure chamber.

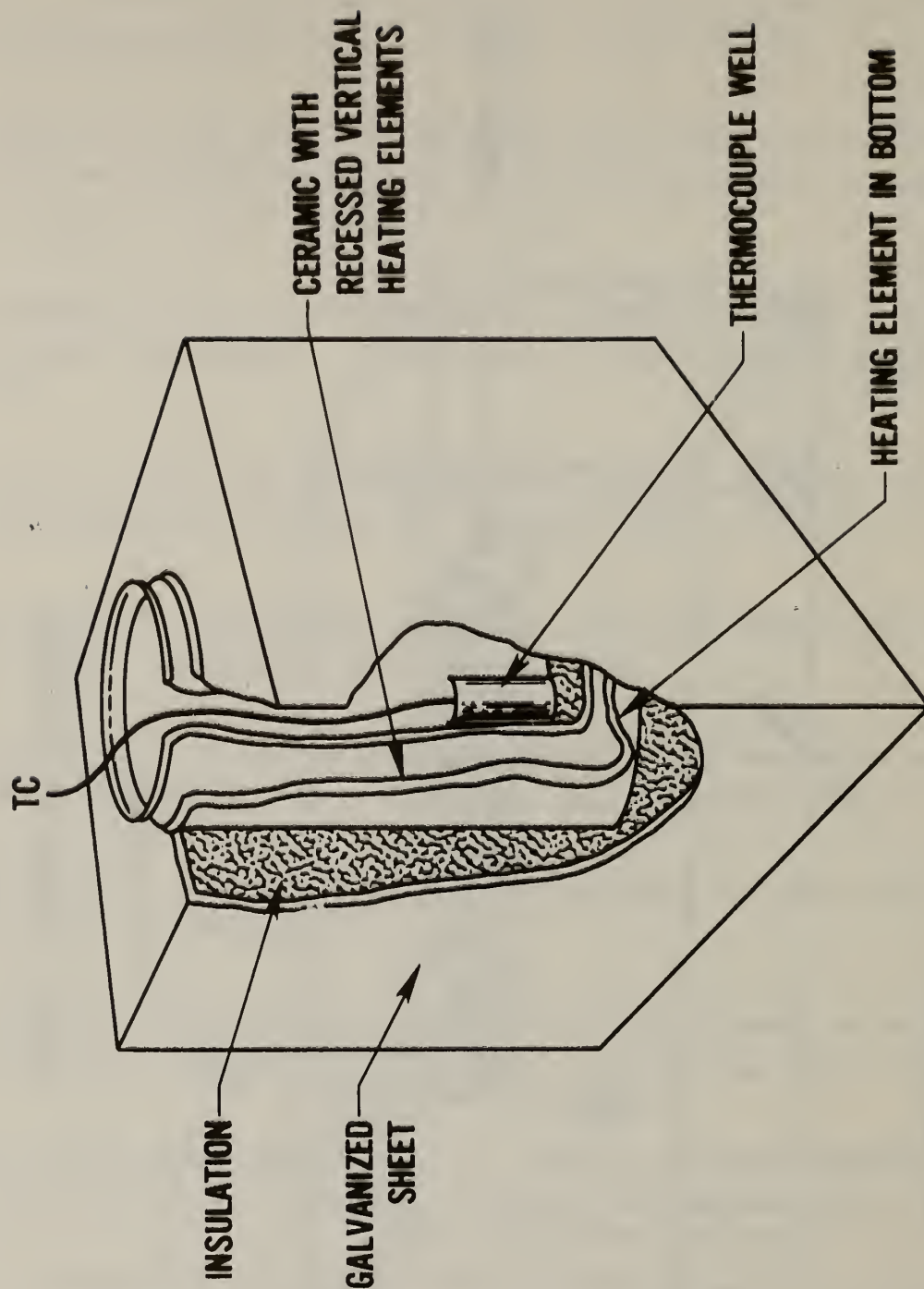


Figure 2. Pyrolysis/combustion furnace.

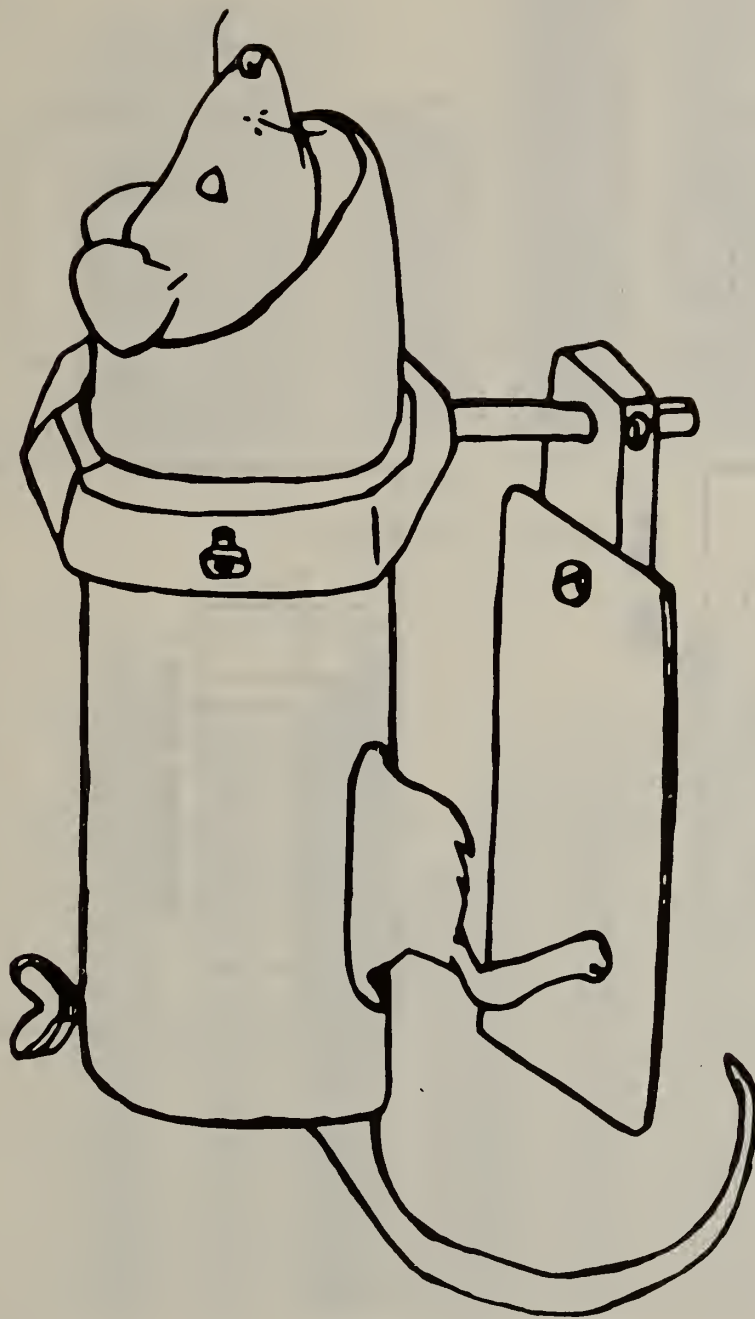


Figure 3. Animal restrainer.

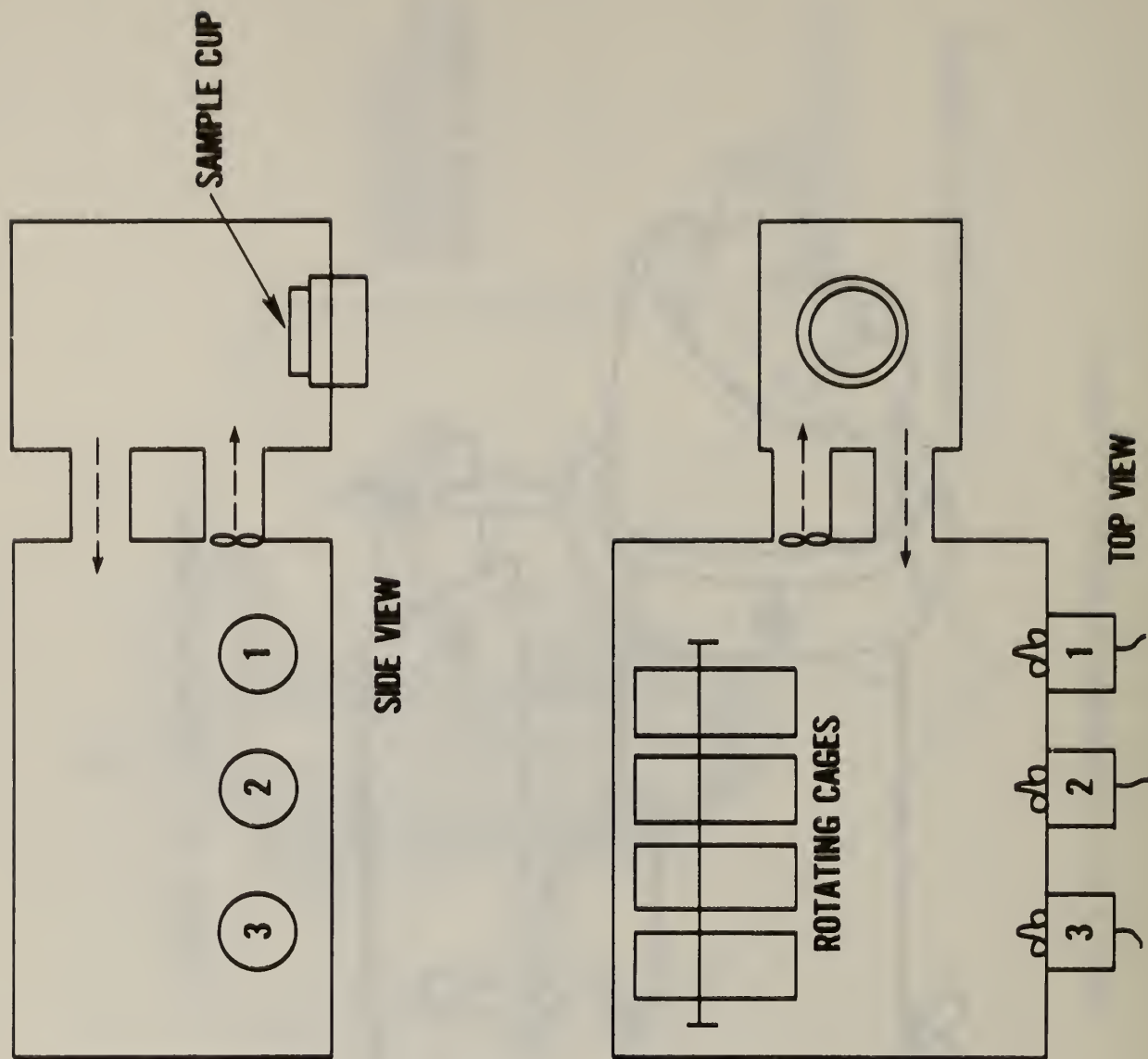


Figure 4. Exposure chamber with separate furnace.

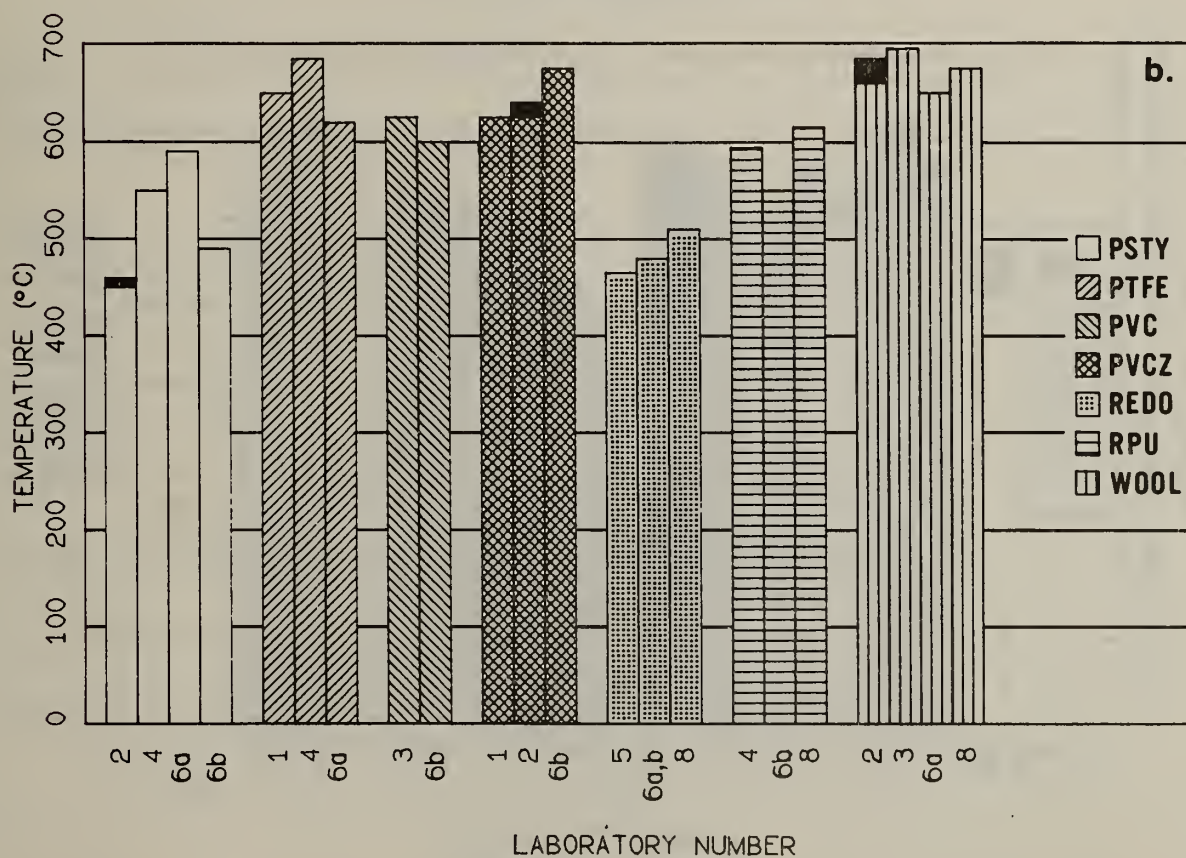
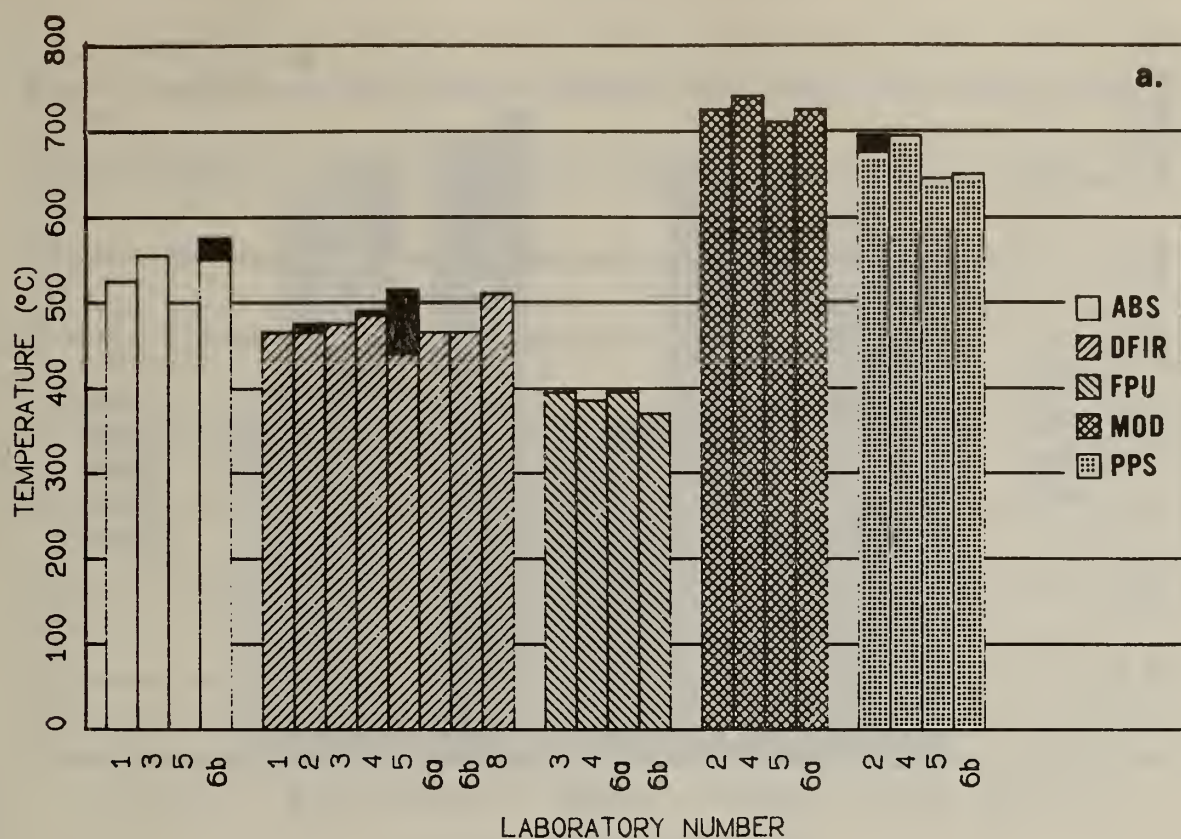


Figure 5a & 5b. Auto-ignition temperatures determined for each material by each laboratory. (Solid black portion of some bars indicates intralaboratory variability.)

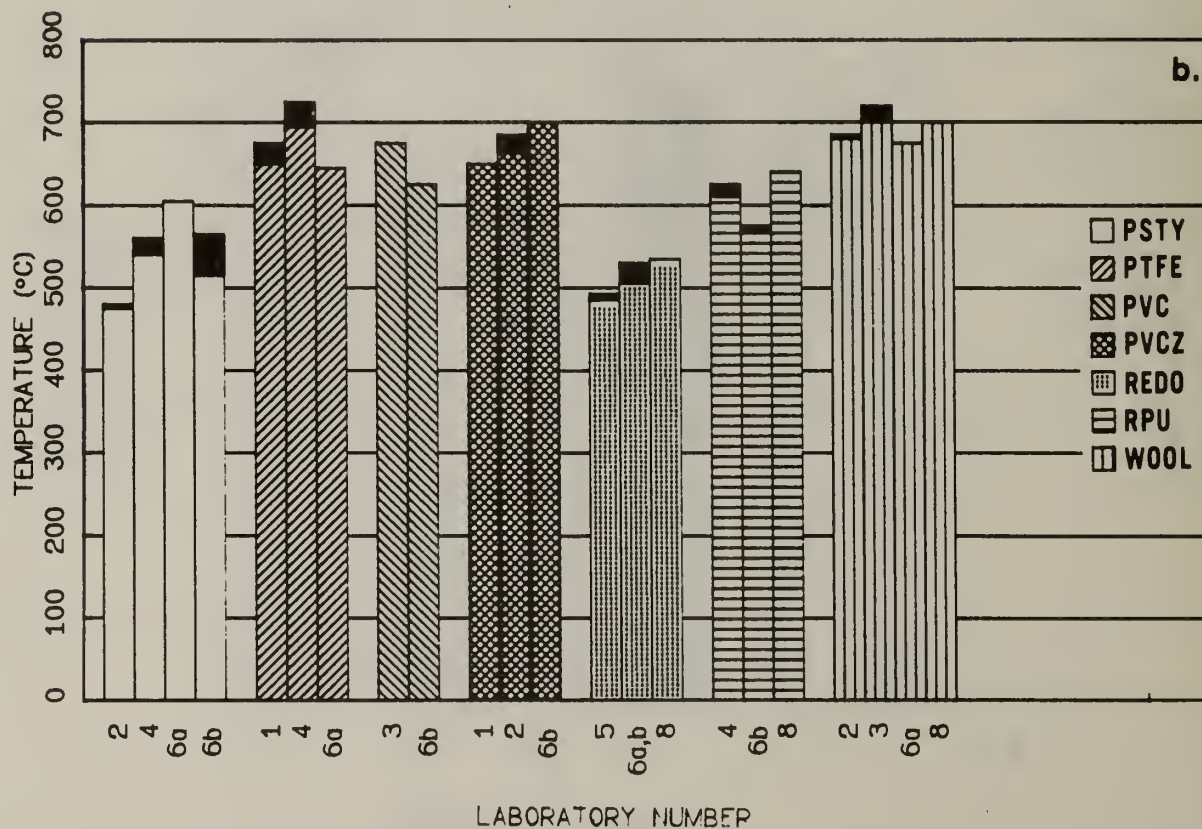
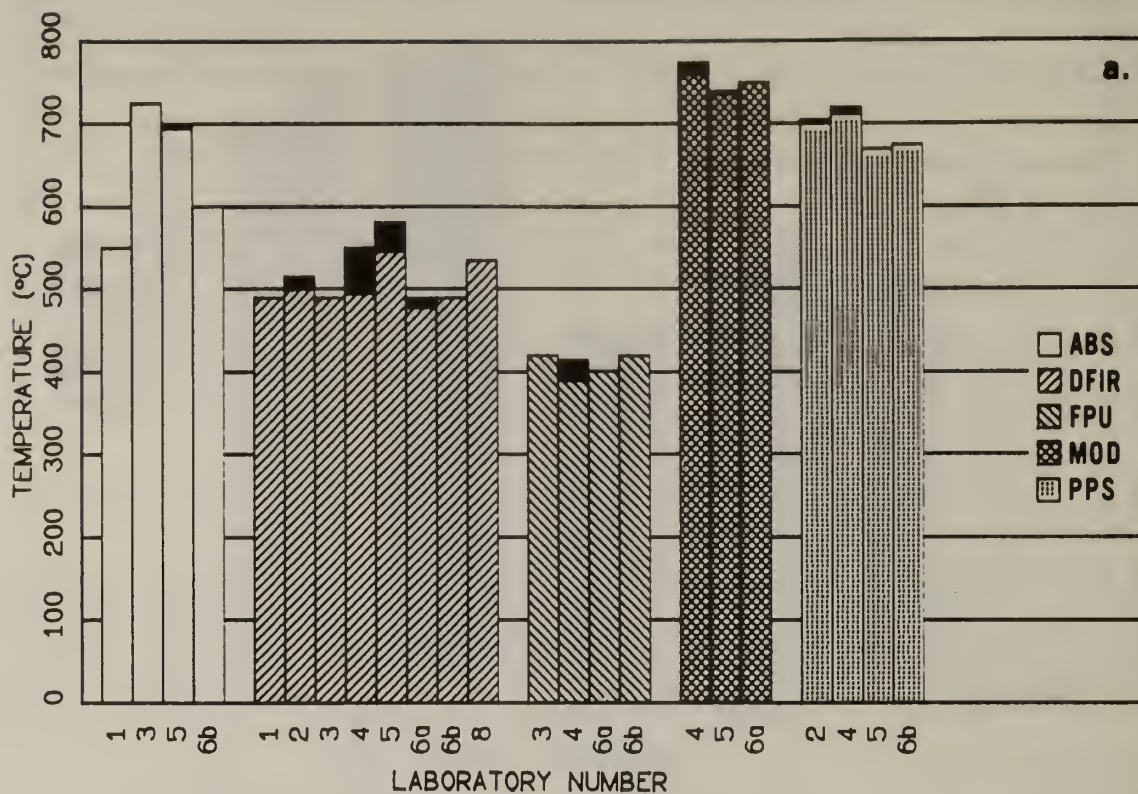


Figure 6a & 6b. Flaming temperatures determined for each material by each laboratory. (Solid black portion of some bars indicates intralaboratory variability.)

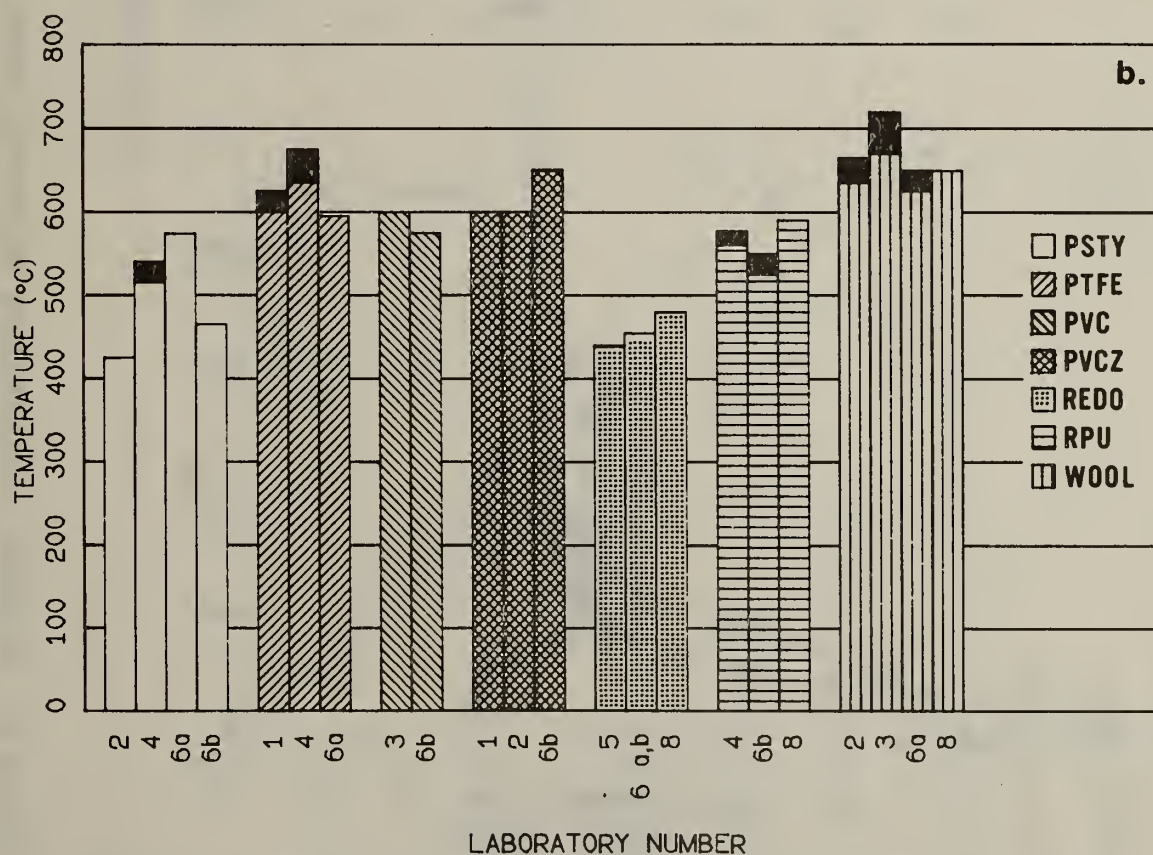
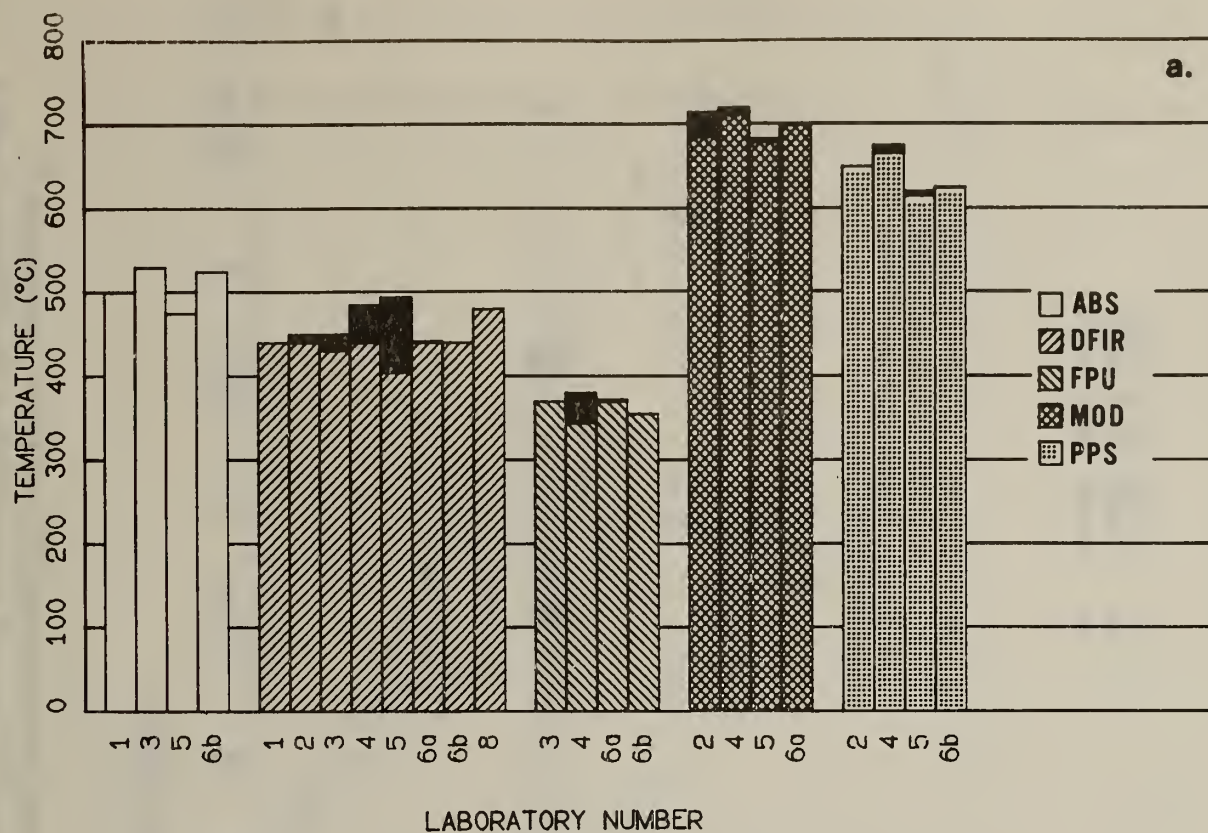


Figure 7a & 7b. Non-flaming temperatures determined for each material by each laboratory. (Solid black portion of some bars indicates intralaboratory variability.)

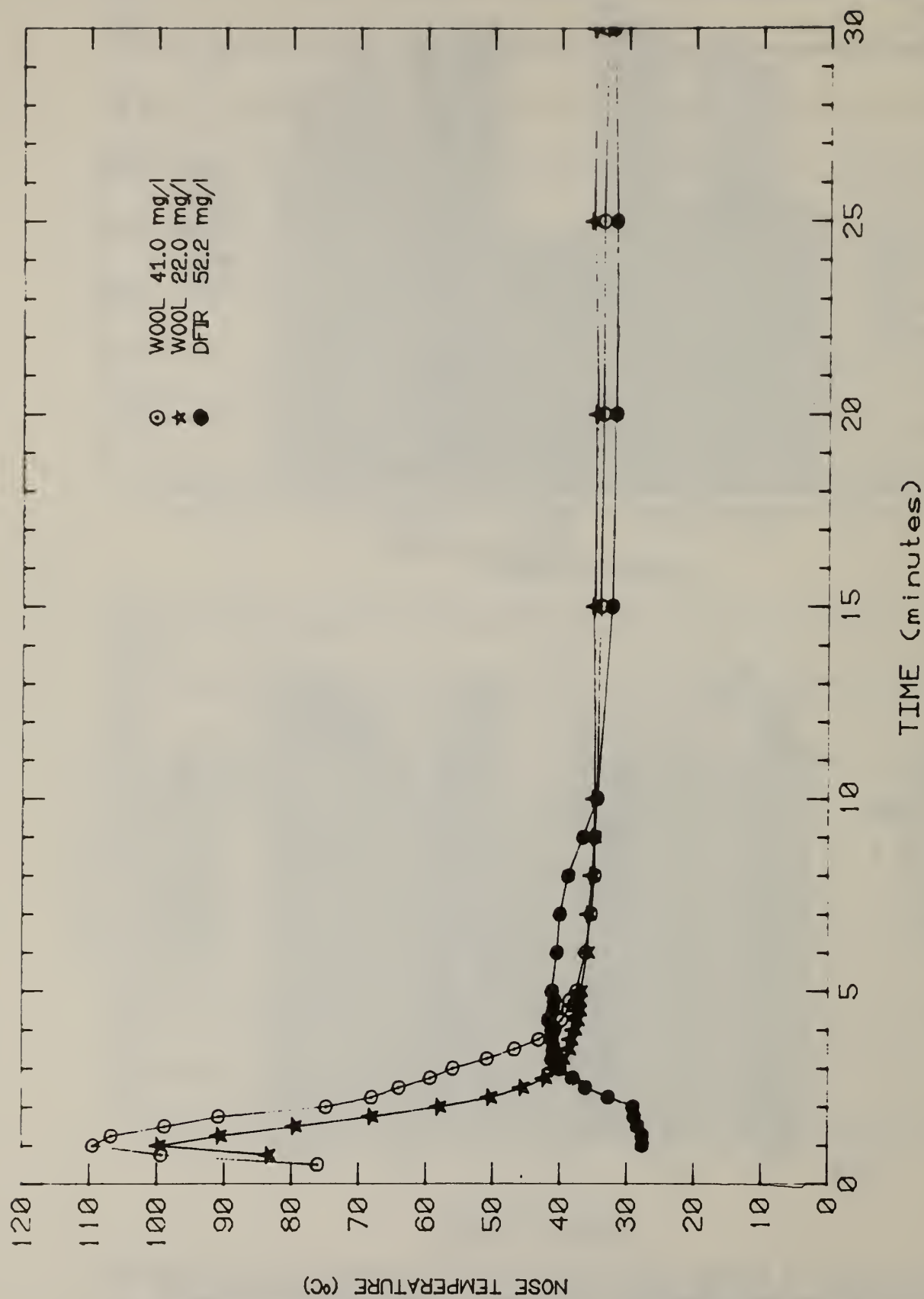


Figure 8. Chamber temperature at animal nose positions during 30 minute exposure to flaming wool and Douglas fir. (NBS data. Furnace temperature for flaming wool was 675°C and for flaming Douglas fir was 490°C.)

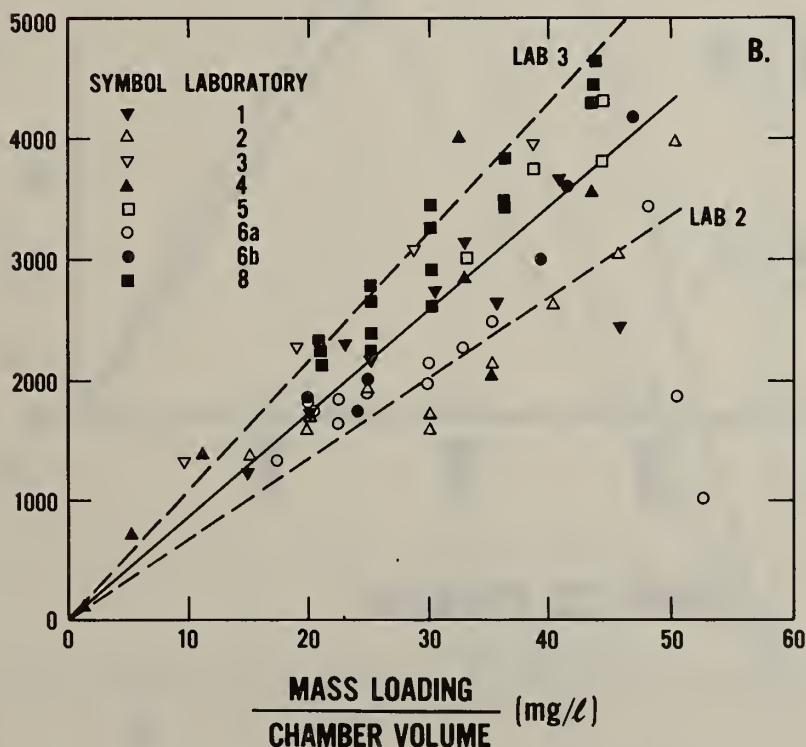
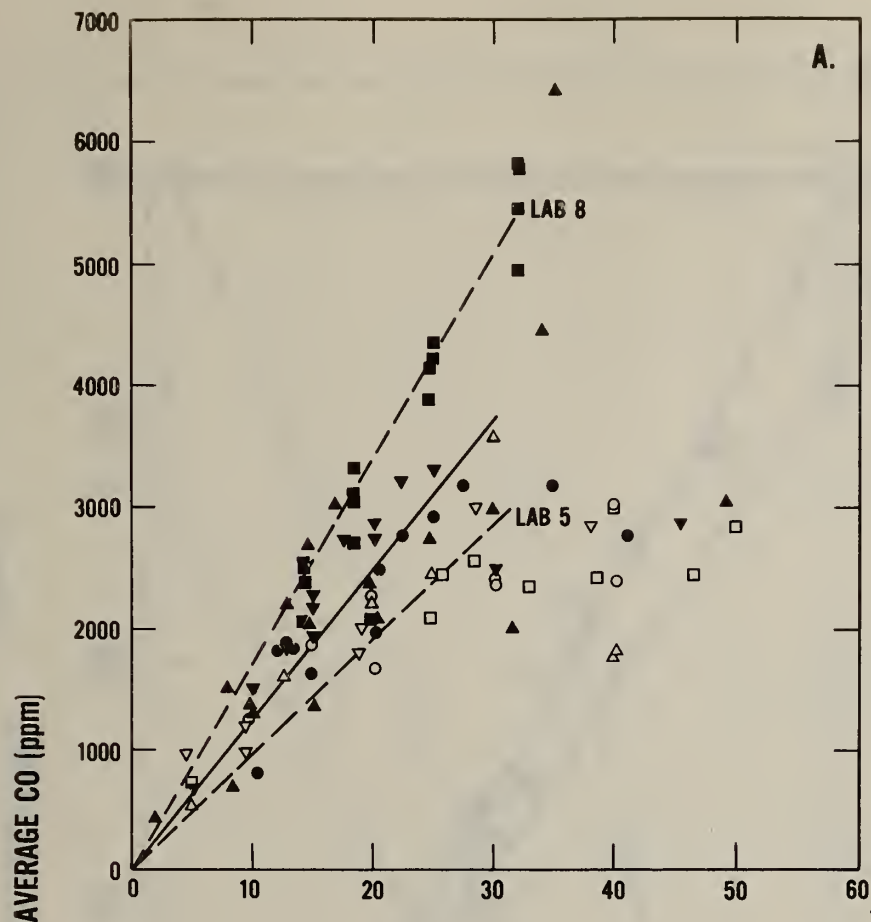


Figure 9. Average carbon monoxide concentrations (ppm) from Douglas fir decomposition as a function of the mass loading/chamber volume (mg/l). A. - Non-flaming results. B. - Flaming results. Solid line - Slope of laboratory results up to 30 mg/l (A) or 50 mg/l (B). Dashed lines - Laboratories with greatest and smallest slopes.

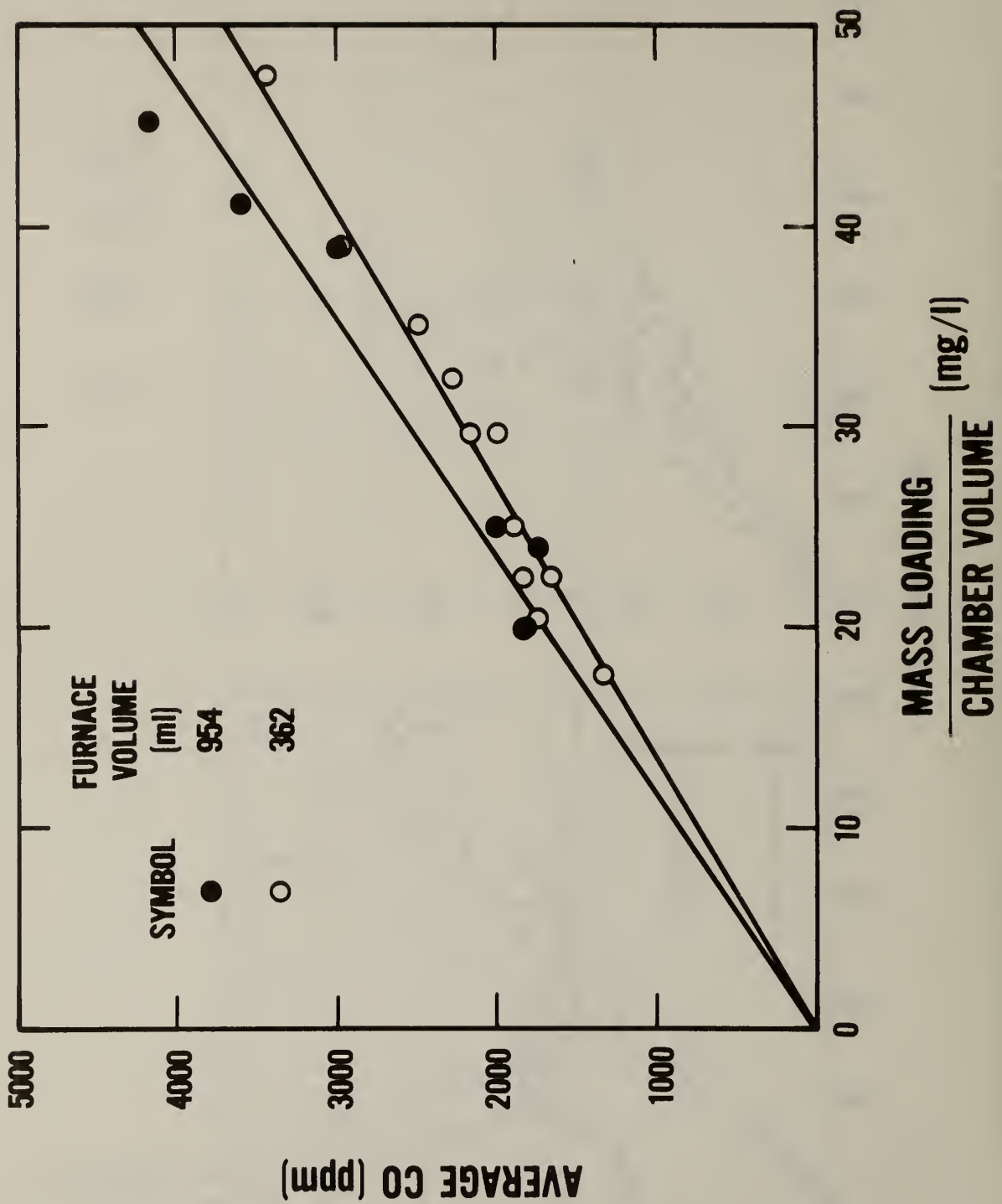


Figure 10. Carbon monoxide generation from Douglas fir decomposed in the flaming mode in both a large and small cup furnace (NBS data).

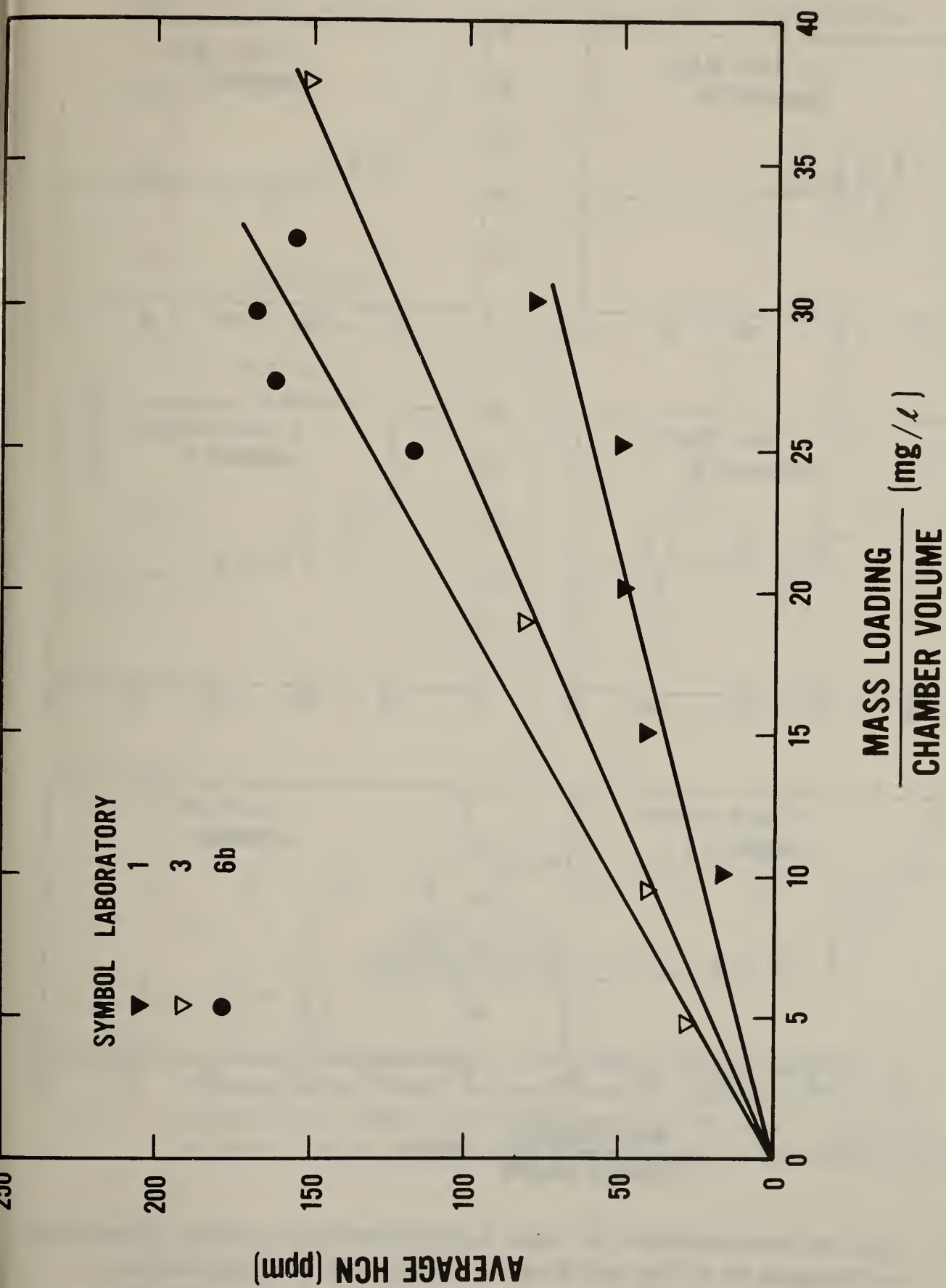


Figure 11. Average hydrogen cyanide concentrations (ppm) from the non-flaming decomposition of ABS as a function of the mass loading/chamber volume (mg/l).

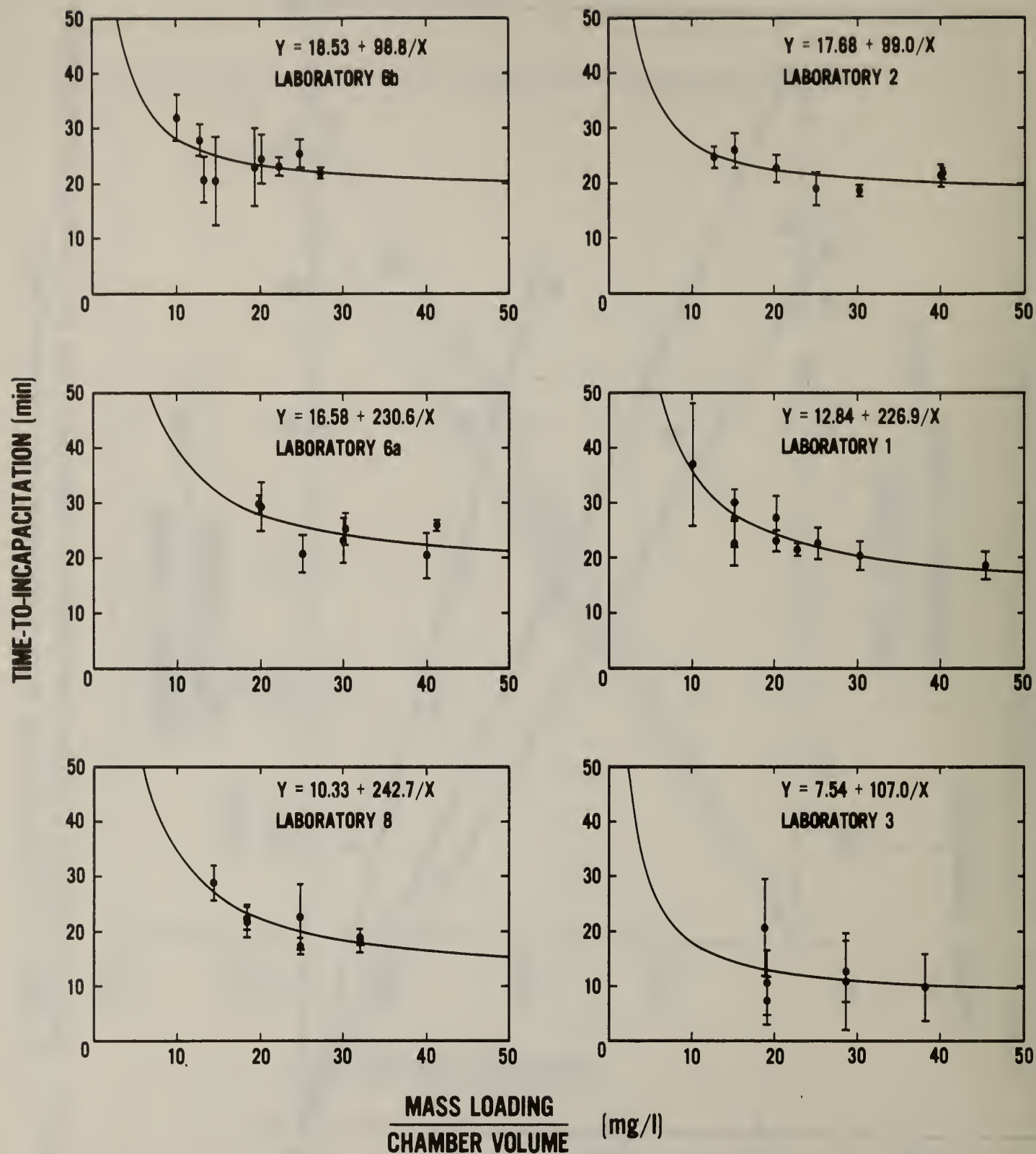


Figure 12. Time-to-incapacitation vs mass loading/chamber volume hyperbolas for Douglas fir in the non-flaming mode from six laboratories. (Symbols refer to the mean and standard deviation of the times-to-incapacitation of the 6 animals tested at each mass loading.)

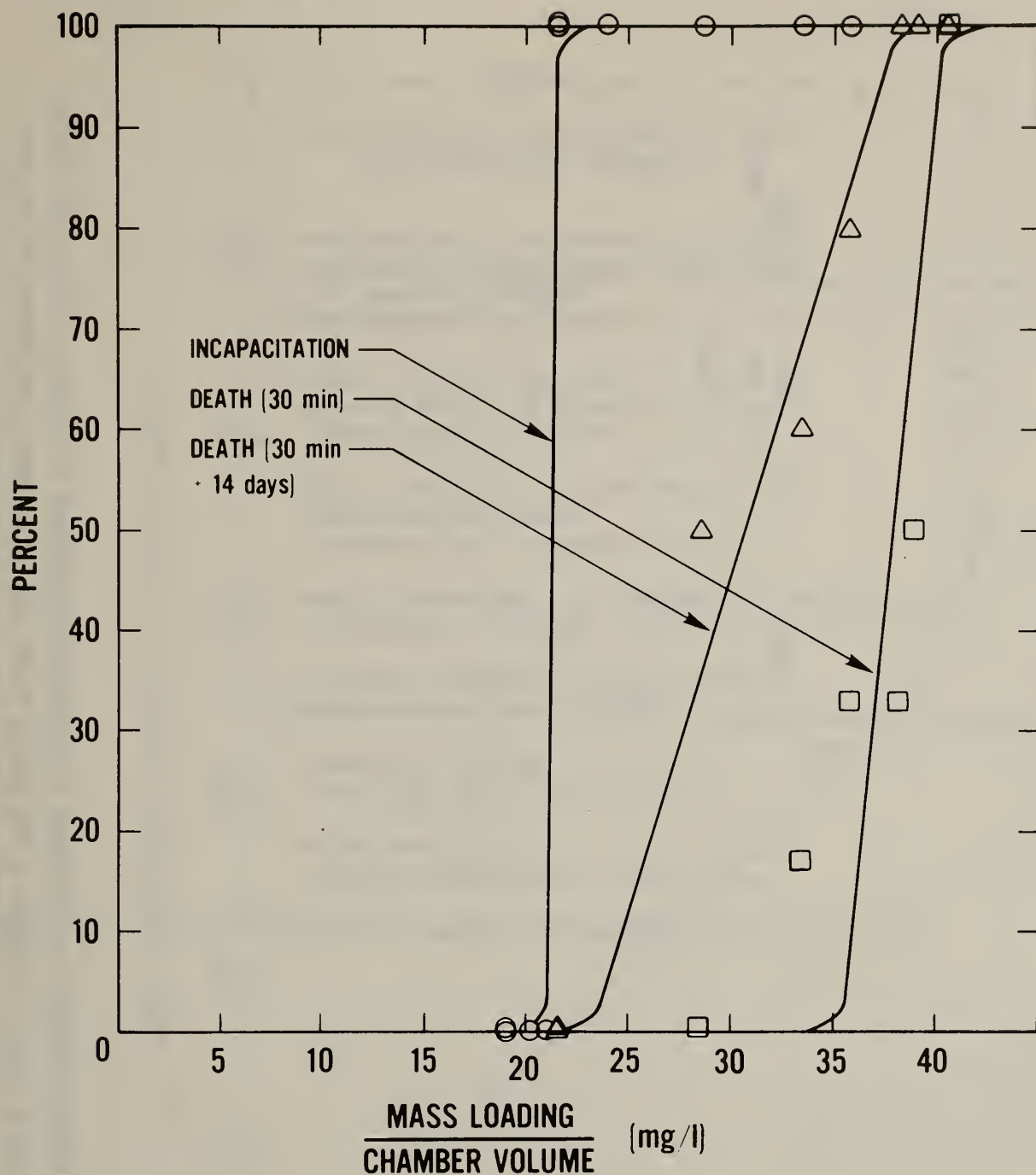


Figure 13. Concentration-response curves resulting from animal incapacitation, death within exposure (30 min.) and death both within and post-exposure (30 min. + 14 days) from the thermal decomposition of wool in the flaming mode.

DETERMINATION OF COHb AT EC50 AND LC50

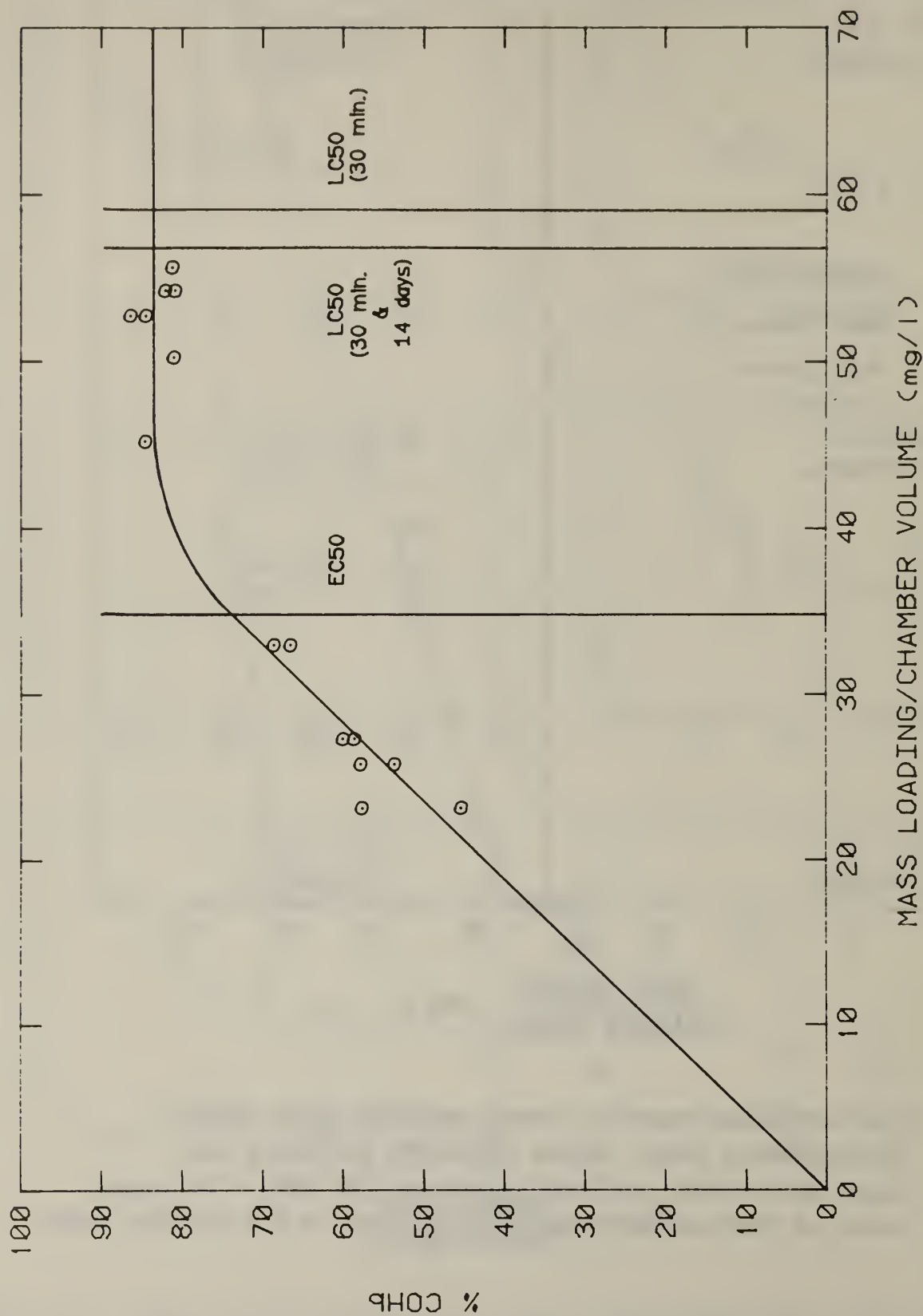


Figure 14. Determination of percent carboxyhemoglobin at EC50, LC50 (30 min.), and LC50 (30 min. + 14 days) from blood values obtained at various mass loadings. (NBS data from flaming red oak.)

Table 1

Laboratories that Participated in the
Interlaboratory Evaluation

- o Armstrong World Industries, Inc.*
Lancaster, Pennsylvania 17604, and
B.F. Goodrich Company*
Cleveland, Ohio 44131
- o Center for Fire Research
National Bureau of Standards
Washington, DC 20234
- o Federal Aviation Administration (FAA)
Aeronautical Center
Oklahoma City, Oklahoma 73125
- o Haskell Laboratory, E.I. DuPont de Nemours & Co.
Wilmington, Delaware 19899
- o Southwest Research Institute (SwRI)
San Antonio, Texas 78284
- o University of Utah
Salt Lake City, Utah 84112
- o Weyerhaeuser Co.
Longview, Washington 98632

*These two laboratories worked together and submitted one set of data.

TABLE 2
List of Materials

Material	Description	Abbreviations
Acrylonitrile butadiene styrene	pellets	ABS
Douglas fir	slabs 10" x 10" x 1"	DFIR
Flexible polyurethane ^{p,a}	flexible foam	FPU
Modacrylic	knit fabric	MOD
Polyphenylsulfone	pellets	PPS
Polystyrene ^{p,b}	rigid foam	PSTY
Polytetrafluoroethylene	powder	PTFE
Poly(vinyl chloride)	pellets	PVC
Poly(vinyl chloride) with zinc ferrocyanide	pellets	PVCZ
Red oak	flooring boards	REDO
Rigid polyurethane ^{p,c}	rigid foam	RPU
Wool	unbleached unwoven fibers	WOOL

p: PRC materials were obtained from the Products Research Committee, Office of Standard Reference Materials, National Bureau of Standards, Washington, D.C. 20234 [15].

a: PRC material, GM-21

b: PRC material, GM-51

c: PRC material, GM-30

It is important to note that the results shown in the following tables pertain to the particular samples tested during this study. The materials used were selected to represent a wide range of properties. No attempt was made to provide statistically valid samples of a given material. Therefore the results should not be used to judge any particular class of material.

Table 3

Experimental Design of Interlaboratory Evaluation

Laboratories	Materials											
	ABS	PTFE	Wool	DFIR	PVCZ	FPU	PSTY	PVC	PPS	RPU	REDO	MOD
1	X	X		X	X							
2			X	X	X		X		X			A
3	X		X	X		X		X				
4		X		X		X	A		X	X		A
5	X			X					X		X	X
NBS	A	X	A	X	A	A	X	X	A	A	X	A
7				N.D.	N.D.			N.D.		N.D.		N.D.
8			X	X						X	X	
9				N.D.		N.D.	N.D.					N.D.

N.D. : Not done

A: Additional material

Table 4a

Standard Laboratory Information

A. Room Conditions

1. Temperature _____ °C
2. Relative Humidity _____ %

B. Furnace

1. Type (e.g., Potts, other) _____
2. Cup Size
 - a. diameter _____ cm
 - b. depth _____ cm
 - c. volume _____ liters

C. Exposure Chamber

1. Dimensions (inside)
 - a. length _____ cm
 - b. width _____ cm
 - c. height _____ cm
 - d. Volume _____ liters

2. Distance from middle of furnace to each animal
(closest to furnace)

a. _____
b. _____
c. _____
d. _____
e. _____
f. _____

(furthest from furnace)

D. Smoke Generation (systems and analysis)

1. Dynamic or static (circle one)
2. Flow rate _____
3. Analytical sampling rate _____
4. Analytical instruments (specify brand and type)
 - a. O₂
 - b. CO
 - c. CO₂
5. How instruments under #4 were calibrated
 - a. _____
 - b. _____
 - c. _____

E. Animal Information

1. Strain and sex _____ (_____)
2. Age (approx.) _____ mo.
3. Number exposed per experiment _____
4. Observation period before use _____ days
5. Exposure mode (head only, full body, other) _____
6. Incapacitation model (hind leg flexure, other) _____
shock current _____ ma
7. Animal retainer material (plexiglas, aluminum, other) _____
8. Blood analysis instrumentation (specify brand or method) _____

Codes:

Smoke Density: None (N); Transparent (TP);
Translucent (TL); Opaque (O)

Smoke Color: White (W); Yellow (Y);
Gray (G); Black (B)

Lethality:

Died during experiment = E; Sacrificed during experiment = S-E;
Sacrificed post exposure = S-PE; Cannulated = C; Died overnight,
discovered in morning = O; Died during morning = A.M.; Died
during afternoon = P.M.; Died over weekend, discovered Monday
morning = W; Lived 14 days = L-14.
(If use symbols - O, W, AM or PM - be sure to include date)
("C" can be used in conjunction with other symbols)

Sample form: Solid, pellets, granular, powder, other _____

Sample conditioning: Temp. _____ °C
Rel. Hum. _____ %
Time _____ hours, days _____

Experimental Information
for Material _____
Identification # _____
Laboratory I.D. # _____

Auto-ignition Temp. $^{\circ}\text{C}$
Non-flaming or Flaming experiments (circle one)
If flaming; ethanol used (Y;N), Ignition spark (Y;N)

[illegible]

Animal Information

48

Table 5

Measurement Techniques for Carbon Monoxide, Carbon Dioxide, and Oxygen

Laboratory	Carbon Monoxide and Carbon Dioxide				Oxygen	
	Frequency of Measurement	Gas Flow Rate from Exposure Box (liters/min)	Type of Instrument	Name of a. Instrument	Type of Instrument	Name of a. Instrument
1	Continuous	0.75	NDIR	Beckman 864	Paramagnetic	Beckman D2
2	Continuous	0.50	NDIR	Beckman 865	Polarographic	Beckman OM11
3	Intermittent (every 3 min)	--	GC	GOW-MAC	Chromatographic	GOW-MAC
4	Continuous	1.0	NDIR	Infrared Industries	Galvanic	Biomarine Industries
5	Continuous	--	NDIR	Beckman 865 (CO) Beckman 864 (CO ₂)	Polarographic	Beckman OM11
6	Continuous	2.0	NDIR	Beckman 865	Galvanic	Bachrach 514-010
8	Intermittent (every 5 min)	--	GC	Carle 8000	Polarographic	Beckman OM11

a. Certain commercial equipment, instruments, or materials are identified in this paper in order to adequately specify the experimental procedure. In no case does such identification imply recommendation or endorsement by the National Bureau of Standards, nor does it imply that the equipment or material identified is necessarily the best available for the purpose.

NDIR: Nondispersive infrared.

GC: Gas chromatograph.

Table 6
Modifications of the Experimental Procedure

Laboratory	Furnace			Exposure Chamber		Strain	Age (months)	#Exposed Test	Animal Information				
	Type	Diameter (cm)	Depth (cm)	Volume (ml)	L x W x H (cm)				Volume (liters)	Pre-exposure Observation (days)	Cannulation (#)	Shock Current (ma)	Restraint Material
1	Potts	5.5	12.7	300	121.9 x 35.6 x 45.7	198	Sprague-Dawley	-	6	7	No	5	plastic
2	Potts	5.0	12.0	236	121.9 x 35.6 x 45.7	201	Fischer 344	4	6	14	No ^c	3	aluminum
3	Potts	5.5	12.0	285	130 x 35.4 x 42	193	Long Evans	3-4	6	14	Yes (3) ^d	1-3	plastic
4	Potts	5.6	12.5	308	120 x 33.5 x 44	177	Sprague-Dawley	2	6	10	No ^e	1-10	plastic
5	Potts	5.5	11.7	278	119.4 x 35.5 x 43.2	182	Sprague-Dawley	2-3	6	5-10	Sometimes (1-2)	4-6	aluminum
6a	Potts	6.1	12.4	362	121.9 x 35.6 x 45.8	199	Fischer 344	2	6	>10	Yes (2) ^f	12.5-13.5	aluminum
6b	Thermocraft	9.0	15.0	954	119.4 x 35.6 x 45.7	194	Fischer 344	2	6	>10	Yes (2) ^f	12.5-13.5	aluminum
8	Other	10.0	1.0	79	75 x 60.8 x 44.8 33.6 x 28.4 x 45.3 12.8 x 7.5 (diam.)	249	Sprague-Dawley	2	3	8	No	8	plastic

- a: NBS smaller furnace
b: NBS larger furnace
c: Blood samples via cardiac puncture, open chest.
d: Cannulated animals kept through 14 day post-exposure period.
e: Blood Samples via cardiac puncture.
f: Cannulated animals sacrificed following exposure.

Table 7
Autoignition, Flaming and Non-flaming Furnace Temperatures

Material	Laboratory	Temperatures (°C)								
		Autoignition	Mean*	S.D.*	Flaming	Mean*	S.D.*	Non-flaming	Mean*	S.D.*
ABS	1	525			550			500		
	3	555			725			530		
	5	500			694-700			475		
	6b	550-575	533	25	600	642	81	525	508	25
DFIR	1	465			490			400		
	2	465-475			500-515			440-450		
	3	475			490			430-450		
	4**	490			495-550			440-485		
	5	440-515			546-581			404-494		
	6a	465			478-490			440-442		
	6b	465			490			440		
	8	510	472	21	535	503	24	480	439	21
FPU	3	395			420			370		
	4**	385			390-415			344-380		
	6a	395			402			370-372		
	6b	370	386	12	420	408	15	355	360	13
MOD	2	725						685-715		
	4**	740			760-775			710-720		
	5	710			736-740			679-683		
	6a	725	725	12	750	749	12	699-701	693	14
PPS	2	675-695			700-705			650		
	4**	694			712-720			665-675		
	5	645			670			615-620		
	6b	650	666	23	675	689	20	624-625	639	23
PSTY	2	450-460			475-480			425		
	4	550			540-560			515-540		
	6a	590			605			574		
	6b	490	520	62	515-565	534	55	465	495	64
PTFE	1	650			650-675			600-625		
	4**	685			695-725			635-675		
	6a	620	652	33	645	663	28	595	610	22
PVC	3	625			675			600		
	6b	600	613	18	625	650	35	575	588	18
PVCZ	1	625			650			600		
	2	625-640			665-685			600		
	6b	675	642	29	700	672	26	649-651	616	28
REDO	5	465			485-492			438-440		
	6a,b	480			505-530			455		
	8	510	485	23	535	508	25	480	458	21
RPU	4**	594			610-625			560-577		
	6b	550			570-575			525-550		
	8	615	586	33	640	607	35	590	558	33
WOOL	2	660-685			680-685			635-665		
	3	695			700-720			670-720		
	6a	650			675			625-650		
	8	675	670	20	700	689	13	650	645	20

*The Mean and the Standard Deviation (S.D.) are based on values listed in the preceeding columns. Where a range of values are provided, the lowest value was used in calculating the mean and the standard deviation.

**The autoignition temperature was not given by the laboratory. Value used in table is the calculated mean between highest non-flaming temperature and lowest flaming temperature.

Table 8*

Toxicity of Modacrylic at Different Temperatures

Mode	Temperature (°C)	LC ₅₀ , 30 minutes + 14 days ^a (mg/l)
Flaming	760 - 775	7.1 (6.4 - 7.9) ^b
Non-Flaming	710 - 720	7.8 (6.3 - 9.7)
	445 - 460	10.0 (6.9 - 14.4)
	390 - 400	13.6 (10.7 - 17.3)
	295 - 305	21.8 (18.4 - 25.8)
	250 - 260	~23.8 ^c [17.0 ^d - 28.3 ^e]
	200	>22.6 ^f

a: Data from laboratory 4

b: 95% confidence limits

c: Approximate value determined from values in brackets

d: 0/6 animals died at this mass loading/chamber volume

e: 5/6 animals died at this mass loading/chamber volume

f: No animals died at this mass loading/chamber volume

*: In this table and subsequent tables, the numbers quoted are as calculated from the data provided by various laboratories. The 95% confidence limits reflect only statistical variations.

Table 9

Maximum Chamber Temperatures Experienced by Animals*

Material	Laboratory	Flaming Mode		Non-Flaming Mode		440°C Mode	
		Temperature (°C)	Mass Loading Chamber Volume (mg/l)	Temperature (°C)	Mass Loading Chamber Volume (mg/l)	Temperature (°C)	Mass Loading Chamber Volume (mg/l)
ABS	1		N.I.		N.I.		N.I.
	3		N.I.		N.I.		N.I.
	5	33	35.0	29	40.0	26	37.6
	6b	40	19.0	32	30.0		N.D.
DFIR	1		N.I.		N.I.		H
	2	40	45.3		N.I.		H
	3	32	38.4		N.I.		H
	4	41	5.6	35	17.0		H
	5		N.I.		N.I.		H
	6a	42	52.2	31	40.2		H
	6b	37	46.4	31	27.5		H
	8		N.I.		N.I.		H
FPU	3		N.I.		N.I.		H
	4		N.I.		N.I.		H
	6a	47	25.1	30	47.7		H
	6b	64	37.5	33	20.0		H
MOD	2		N.D.	37	5.0		N.D.
	4	40	6.2	38	5.5	34	11.7
	5	40	6.0	36	10.0	31	6.0
	6a	37	5.5	38	3.3	28	4.7
	6b		N.D.		N.D.	33	5.3
PPS	2	43	26.2	39	18.7	30	19.9
	4	37	39.6	49	9.9	34	8.8
	5	31	18.5	32	12.0	24	40.0
	6b	53	20.0	39	10.0		N.D.
PSTY	2	45	39.9	30	50.1		H
	4	35	17.1		N.I.		H
	6b	56	40.0	36	34.1		H
PTFE	1		N.I.		N.I.		N.I.
	4		N.I.	36	0.05		N.I.
	6a		N.I.		N.I.		N.D.
PVC	3		N.I.		N.I.		N.I.
	6b	37	30.0	35	18.0	35	20.0
PVCZ	1		N.I.		N.I.		N.I.
	2	43	19.9	40	11.2	30	12.4
	6b	37	12.0	40	12.0	33	12.5
REDO	5	34	60.0	30	45.0		H
	6a	46	50.2	29	45.2		H
	6b	37	25.8		N.D.		H
	8		N.I.		N.I.		H
RPU	4	29	0.1	25	33.9	32	0.1
	6b	40	20.0	31	41.1		N.D.
	8		N.I.		N.I.		N.I.
WOOL	2	50	39.9	34	40.1	31	27.2
	3		N.I.		N.I.		N.I.
	6a	109	41.0	36	17.6		N.D.
	6b		N.I.		N.D.	36	25.1
	8		N.I.		N.I.		N.I.

*Measured at nose position of animals.

a NBS small furnace

b NBS large furnace

N.I. no information provided

N.D. not done

H not determined as non-flaming temperatures were less or within 50°C of 440°C.

Table 10

Highest Average Chamber Temperatures Experienced by Animals*

Material	Laboratory	Flaming Mode		Non-Flaming Mode		440°C Mode	
		Temperature (°C)	Mass Loading Chamber Volume (mg/l)	Temperature (°C)	Mass Loading Chamber Volume (mg/l)	Temperature (°C)	Mass Loading Chamber Volume (mg/l)
ABS	1		N.I.		N.I.		N.I.
	3		N.I.		N.I.		N.I.
	5	31	20.0	28	40.0	25	37.6
	6b	32	19.5	30	25.0		N.D.
DFIR	1		N.I.		N.I.		H
	2		N.I.		N.I.		H
	3		N.I.		N.I.		H
	4	39	11.3	35	17.0		H
	5		N.I.		N.I.		H
	6a	33	52.2	31	30.4		H
	6b	30	46.4	29	27.5		H
	8	34	36.2	33	32.1		H
FPU	3		N.I.		N.I.		H
	4		N.I.		N.I.		H
	6a	30	25.1	29	47.7		H
	6b	36	37.5	29	20.0		H
MOD	2		N.D.	35	5.0		N.D.
	4	39	6.2	38	7.1	32	11.7
	5	37	6.0	34	10.0	30	6.0
	6a	35	5.4	36	3.3	27	4.7
	6b		N.D.		N.D.	31	5.3
PPS	2	38	30.0	37	18.7	28	19.8
	4	37	1.0	47	9.9	33	8.8
	5	29	18.5	30	12.0	23	40.0
	6b	41	20.0	37	10.0		N.D.
PSTY	2	33	34.9	29	35.0		H
	4		N.I.		N.I.		H
	6b	35	40.0	27	34.1		H
PTFE	1		N.I.		N.I.		N.I.
	4	40	0.14	38	.08	33	5.7
	6a		N.I.		N.I.		N.D.
PVC	3		N.I.		N.I.		N.I.
	6b	34	15.0	33	18.0	29	20.0
PVCZ	1		N.I.		N.I.		N.I.
	2	42	19.9	38	11.2	29	12.4
	6b	35	10.0	36	10.0	30	12.5
REDO	5	33	60.0	30	45.0		H
	6a	34	50.2	28	40.2		H
	6b	36	25.8		N.D.		H
	8	37	60.2	34	49.2		H
RPU	4	27	0.1		N.I.	33	20.8
	6b	33	20.0	30	41.1		N.D.
	8	34	12.5	31	29.3	28	35.2
WOOL	2	38	44.9	33	30.2	30	27.2
	3		N.I.		N.I.		N.I.
	6a	38	41.0	34	17.6		N.D.
	6b		N.I.		N.D.	26	25.1
	8	39	77.5	32	21.7	29	35.2

*For explanation of letters, see legend to table 9.

Table 11

Carbon Monoxide Production Per Unit $\frac{\text{Mass Loading}}{\text{Chamber Volume}}$

Material	Laboratory	Flaming Mode			Non-Flaming Mode		
		Mean*	S.D.	n	Mean	S.D.	n
		$\left(\frac{\text{ppm}}{\text{mg/l}}\right)$			$\left(\frac{\text{ppm}}{\text{mg/l}}\right)$		
ABS	1	54	23	8	15	12	5
	3	110	22	3	140	160	5
	5	66	12	3	13	2.1	6
	6b	75	16	7	22	2.5	3
FPU	3	78	80	4	130	83	5
	4	26	14	7	47	22	22
	6a	19	1.8	2	39	18	11
	6b	26	1.5	3	30	19	2
MOD	2		N.D.		35	12	5
	4	86	45	4	52	12	4
	5	100	46	5	76	5.4	5
	6a	77	34	10	82	2.4	6
PPS	2	70	14	6	150	39	7
	4	120	39	4	150	116	3
	5	200	19	5	340	56	5
	6b	180	17	6	470	26	7
PSTY	2	46	1.8	5	2.3	1.1	2
	4	70	5.3	10	13	2.3	4
	6b	34	3.6	4	1.8	0.4	3
PVC	3	115	82	5	65	40	5
	6b	54	19	4	32	5.2	6
PVCZ	1	52	11	6	44	13	7
	2	72	7.4	7	48	9.6	6
	6b	150	12	7	100	10	2
REDO	5	69	6.6	7	87	7.8	5
	6a	57	9.4	7	83	7.5	9
	6b	37	2.4	6		N.D.	
	8	49	3.0	16	96	5.7	16
RPU	4	36	10	2	28	29	2
	6b	130	12	9	45	8.8	11
	8	94	12	16	41	5.0	4

Table 11. Continued

Material	Laboratory	Flaming Mode			Non-Flaming Mode		
		Mean*	S.D.	n	Mean	S.D.	n
		$\left(\frac{\text{ppm}}{\text{mg/l}}\right)$			$\left(\frac{\text{ppm}}{\text{mg/l}}\right)$		
WOOL	2	22	2.6	8	30	7.5	6
	3	68	16	5	69	15	5
	6a	25	4.1	12	39	7.8	8
	6b	25	4.3	2		N.D.	
	8	24	2.2	18	26	6.6	18

*Mean of $\frac{\text{Average gas concentration (ppm) for each 30 minute exposure}}{\text{Mass loading/chamber volume (mg/l)}}$

S.D.: standard deviation

n: number of values used to calculate mean

a: NBS small furnace

b: NBS large furnace

N.D.: not done

Table 12

Carbon Monoxide Production for Douglas Fir in the Flaming Mode

Lab #	1	2	3	4	5	6a	6b	8
	ML CV	Average COD. (ppm)	ML CV	Average COD. (ppm)	ML CV	Average COD. (ppm)	ML CV	Average COD. (ppm)
	(mg/l)	(mg/l)	(mg/l)	(mg/l)	(mg/l)	(mg/l)	(mg/l)	(mg/l)
15.1	1200	15.3	1400	1.0	100	17.6	1300	20.9
20.2	1700	20.0	1600	1.0	80	20.1	1800	20.9
20.2	1700	25.0	1900	5.6	720	20.6	1700	20.9
23.2	2300	30.0	1600	11.3	1400	22.6	1700	20.9
25.2	2200	30.0	1700	32.2	4000	22.7	1800	25.1
30.3	2700	30.0	1700	32.8	2800	25.1	1900	25.1
32.8	3100	35.1	2100	34.5	2000	29.8	2000	25.3
35.3	2600	40.1	2600	43.0	3500	29.8	2200	25.3
40.4	3700	45.3	3100			32.7	2300	30.1
		49.9	4000			35.2	2500	30.1
						39.0	3000	30.1
						47.7	3400	30.1
								36.2
								34.00
								36.2
								38.00
								36.2
								3500
								43.4
								4400
								43.4
								4600
								43.4
								4600
								43.4
								4300
Mean: 87		69	120	98	93	76	83	100
n: 9		10	4	8	4	12	6	20
S.D.: 7.3		12	15	25	5.4	6.7	7.2	7.6
RSD: 8.4		17	13	26	5.8	8.8	8.7	7.6

a: NBS small furnace

b: NBS large furnace

c: $\frac{ML}{CV}$ = Mass Loading
Chamber Volume

d: CO concentration (ppm) averaged over each 30 minute exposure

Mean: Mean of $\frac{\text{Average CO concentration (ppm)}}{\text{Mass loading/chamber volume (mg/l)}}$ for each 30 minute exposure

S.D.: Standard deviation

n: Number of experiments

RSD(%): Relative standard deviation = $\frac{S.D.}{\text{mean}} \times 100$

Table 13
Carbon Monoxide Production for Douglas Fir in the Non-Flaming Mode

[illegible]

*For explanation of superscript letters, see legend to Table 12.

Table 14

Laboratories Statistically Similar on Basis of "w"
Test on Carbon Monoxide Concentrations from Douglas Fir

Mode	w*	Laboratory Number (mean**)							
Non-Flaming	43	5(110)	6a(110)	6b(120)	2(120)	3(120)	1(140)	4(140)	8(170)
Flaming	19	2(69)	6a(76)	6b(83)	1(87)	5(95)	4(98)	8(100)	3(120)

Laboratories connected by a line are similar and laboratories not connected by a line are not similar (i.e. statistically significantly different)

$$*w = \frac{q_{.95} S_e}{n_H}$$

**Mean of $\frac{\text{Average CO concentration (ppm) for each 30 minute exposure}}{\text{Mass loading/chamber volume (mg/l)}}$

$q_{.95}(t, v)$ from Table A-10, reference [9].

t = number of laboratories

$$v = (n_1 + n_2 + \dots + n_8) - t$$

n = number of experiments done by each laboratory

$$S_e = \frac{(n_1 - 1) S_1^2 + (n_2 - 1) S_2^2 + \dots + (n_8 - 1) S_8^2}{(n_1 + n_2 + \dots + n_8) - t} \quad 1/2$$

S = standard deviation

$$n_H = t / \left(\frac{1}{n_1} + \frac{1}{n_2} + \dots + \frac{1}{n_8} \right)$$

(a) NBS small furnace

(b) NBS large furnace

Table 15

Maximum Average Carbon Dioxide Concentrations Produced
During Animal Exposure

Mode	Material	Maximum Average CO ₂ ^a (ppm)	Laboratory ^b	Mass Loading Chamber Volume (mg/l)
<u>Flaming</u>				
	ABS	14000	6b	30.0
	DFIR	42000	6b	46.4
	FPU	46000	6b	37.5
	MOD	17000	5	2.0
	PPS	37000	5	10.0
	PSTY	42000	4	37.4
	PVC	13000	3	38.1
	PVCZ	10000	6b	15.0
	REDO	63000	8	88.4
	RPU	14000	6b	14.0
	WOOL	56000	8	77.7
<u>Non-Flaming</u>				
	ABS	6300	5	40.0
	DFIR	14000	2	30.2
	FPU	7800	4	3.0
	MOD	8100	5	12.0
	PPS	9400	4	26.7
	PSTY	7500	4	32.9
	PVC	5400	3	14.3
	PVCZ	6400	6b	14.0
	REDO	15000	8	49.4
	RPU	8700	8	35.1
	WOOL	7800	6a	25.1

a: CO₂ concentration (ppm) averaged over the 30 minute exposure.

b: Laboratory which found the maximum average concentration.

Table 16

Carbon Dioxide Production Per Unit $\frac{\text{Mass Loading}}{\text{Chamber Volume}}$

Material	Laboratory	Flaming Mode			Non-Flaming Mode		
		Mean*	S.D.	n	Mean	S.D.	n
		$\left(\frac{\text{ppm}}{\text{mg/l}}\right)$			$\left(\frac{\text{ppm}}{\text{mg/l}}\right)$		
ABS	1	500	190	8	230	80	5
	3	530	220	3	530	420	5
	5	440	89	3	150	48	6
	6b	550	58	7	170	15	3
DFIR	1	640	63	9	400	150	11
	2	740	300	10	440	90	7
	3	1000	310	4	490	240	6
	4	990	760	8	330	210	17
	5	730	44	4	200	55	3
	6a	690	83	7	290	66	5
	6b	930	71	6	300	70	9
	8	800	56	20	210	85	16
FPU	3	2000	1000	4	550	500	5
	4	450	220	7	460	730	22
	6a		N.I.		110	53	10
	6b	1200	17	3	130	27	2
MOD	2		N.D.		710	140	3
	4	830	680	4	310	150	4
	5	2600	3300	5	940	410	5
	6a	900	400	10	1000	240	6
PPS	2	800	84	6	380	200	7
	4	1900	2500	4	2100	3200	3
	5	1700	1200	5	580	190	5
	6b	1100	59	3	540	120	5
PSTY	2	880	80	5	50	19	2
	4	1300	570	10	160	50	4
	6b	500	84	4	52	6	3
PVC	3	840	560	5	420	220	5
	6b	320	98	4	230	87	6
PVCZ	1	560	200	6	670	280	7
	2	540	100	7	430	86	6
	6b	650	79	7	470	20	2
REDO	5	790	140	7	300	67	5
	6a	650	62	7	240	43	9
	6b	780	110	6	N.D.		
	8	770	49	16	290	50	16

Table 16. Continued

Material	Laboratory	Flaming Mode			Non-Flaming Mode		
		Mean*	S.D.	n	Mean	S.D.	n
		($\frac{\text{ppm}}{\text{mg/l}}$)			($\frac{\text{ppm}}{\text{mg/l}}$)		
RPU	4	180	75	2	200	100	2
	6b	900	82	4	230	60	2
	8	570	79	16	230	22	4
WOOL	2	700	54	8	200	43	6
	3	1400	500	5	570	310	5
	6a		N.I.		280	90	8
	6b		N.I.		N.D.		
	8	819	120	18	160	28	18

*For explanation of superscript symbols and letters, see legend to table 11.

Table 17

Minimum Average Oxygen Concentrations*

Material	Laboratory	Oxygen Concentration (%)		440°C
		Flaming Mode	Non-Flaming Mode	
ABS	1	20.1	20.7	21.0
	3	20.4	20.7	20.8
	5	20.3	20.1	20.2
	6b	18.9	20.1	N.D.
DFIR	1	19.0	20.0	H
	2	N.I.	N.I.	H
	3	18.2	20.5	H
	4	18.1	19.8	H
	5	17.8	19.9	H
	6a	17.8	19.9	H
	6b	16.3	19.2	H
	8	19.1	19.6	H
FPU	3	16.4	20.6	H
	4	N.I.	N.I.	H
	6a	17.4	19.3	H
	6b	14.2	19.9	H
MOD	2	N.D.	N.I.	N.D.
	4	N.I.	N.I.	N.I.
	5	19.7	19.8	20.1
	6a	18.7	18.5	20.3
	6b	N.D.	N.D.	19.9
PPS	2	N.I.	N.I.	20.3
	4	19.5	N.I.	N.I.
	5	18.2	19.8	20.5
	6b	17.7	19.7	N.D.
PSTY	2	N.I.	N.I.	H
	4	N.I.	N.I.	H
	6b	17.9	20.2	H
PTFE	1	20.1	21.0	21.0
	4	19.9	19.8	20.0
	6a	N.I.	N.I.	N.D.
PVC	3	19.5	20.6	N.I.
	6b	19.2	19.3	19.7
PVCA	1	20.6	20.9	20.8
	2	N.I.	18.3	N.I.
	6b	19.3	19.9	19.5
REDO	5	16.6	18.8	H
	6a	17.2	19.6	H
	6b	16.8	N.D.	H
	8	17.9	19.2	H
RPU	4	N.I.	N.I.	N.I.
	6b	18.8	19.7	N.D.
	8	20.4	20.1	20.3
WOOL	2	17.3	17.5	N.I.
	3	16.7	19.3	20.7
	6a	16.2	20.7	N.D.
	6b	17.5	N.D.	19.8
	8	19.5	19.7	20.7

*Concentration of O₂ from the experiment which produced the lowest average O₂ concentration over the 30 minute exposure.

a: NBS small furnace

b: NBS large furnace

N.I.: No information provided

N.D.: Not done

H: Not done as non-flaming temperatures were less or within 50°C of 440°C

Table 18

Average Hydrogen Cyanide Production for Nitrogen Containing Materials
Decomposed Under Flaming and Non-Flaming Conditions

Material	Laboratory	Flaming Mode		Non-Flaming Mode	
		Mass Loading	Average*	Mass Loading	Average*
		Chamber Volume (mg/ l)	HCN (ppm)	Chamber Volume (mg/ l)	HCN (ppm)
ABS	1	10.1	81	10.1	16
		15.1	120	15.1	42
		16.6	140	20.2	49
		20.2	150	25.2	50
		25.2	170	30.3	78
	3	4.8	44	4.8	28
		9.5	100	9.5	41
		14.3	160	19.0	81
		19.0	230	38.0	150
	6b	17.5	91	25.0	120
		18.5	84	27.5	160
		19.0	120	30.0	170
		19.5	44	32.5	160
		20.0	140		
		20.0	190		
		30.0	250		
FPU	3	5.0	11	2.5	4
		9.6	22	4.8	3
		18.9	7	9.6	3
		38.0	32	19.1	8
				37.9	8
	6a, b	25.1	23	15.1	9
		30.0	19	20.0	8
		31.2	26	20.1	19
		35.0	15	25.1	17
		37.5	14	29.6	21
		40.0	17	30.0	9
				32.2	10
				40.2	6
				42.7	19
				42.7	16
				45.2	12
				47.7	17
				47.7	17
MOD	6a	2.5	110	2.8	120
		3.0	110	3.3	130
		3.3	110	3.8	200
		3.5	160	4.5	220
		3.8	170	5.0	260
		4.4	180	7.0	320

Table 18. Continued

Material	Laboratory	Flaming Mode		Non-Flaming Mode	
		Mass Loading	Average*	Mass Loading	Average*
		Chamber Volume (mg/ℓ)	HCN (ppm)	Chamber Volume (mg/ℓ)	HCN (ppm)
MOD	6a	5.0	208		
		5.4	260		
		5.5	250		
		6.2	220		
PVCZ	1	5.0	50	4.0	45
		10.1	92	5.0	63
		12.6	111	10.1	130
		15.1	158	12.6	210
				15.1	180
	6b	12.0	90	10.0	120
		14.0	100	12.1	140
		15.0	140	14.0	220
		15.5	100		
		16.0	110		
		17.0	140		
	6b	7.5	66	20.0	59
		10.4	68	20.7	15
		13.0	130	21.4	12
		14.0	170	30.2	8
		14.9	150	39.6	61
		20.0	190	39.8	100
				39.9	40
WOOL	3			40.0	19
				41.1	17
		9.7	30	4.8	120
		28.3	130	9.5	310
	6a	28.6	280	14.3	250
		28.6	480	19.0	120
				23.8	230
		20.1	70	12.6	160
		21.4	70	17.6	320
		22.0	80	22.6	330
		22.6	50	25.1	250
		22.6	70	30.2	200
		25.1	120	32.7	210
		30.2	110		
		30.2	120		
		35.2	150		
		37.7	190		
		41.0	310		
		42.7	200		

*Average HCN concentration (ppm) for each 30 minute exposure

a: NBS small furnace

b: NBS large furnace

Table 19

Hydrogen Cyanide Production Per Unit $\frac{\text{Mass Loading}}{\text{Chamber Volume}}$

Materials	Laboratory	Flaming Mode			Non-Flaming Mode		
		Mean*	S.D.	n	Mean	S.D.	n
		$\left(\frac{\text{ppm}}{\text{mg/l}}\right)$			$\left(\frac{\text{ppm}}{\text{mg/l}}\right)$		
ABS	1	7.8	0.6	5	2.4	0.7	5
	3	11	1.2	4	4.6	0.8	4
	6b	6.4	2.6	7	5.3	0.5	4
FPU	3	1.4	1.0	4	0.6	0.6	5
	6a, b	0.6	0.2	6	0.4	0.2	13
MOD	6a	41	5.3	10	47	4.8	6
PVCZ	1	9.6	0.8	4	13	2.0	5
	6b	7.6	1.0	6	13	2.2	3
RPU	6b	9.5	1.8	6	1.2	1.0	9
WOOL	3	9.1	5.7	4	18	11	5
	6a	4.1	1.4	12	12	4.7	6

*For explanation of letters and superscripts, see legend to table 11.

TABLE 20

Incapacitation Times After Exposure to Non-Flaming PVC

Mass Loading Chamber Volume (mg/l)	Test Duration (min)	Animal Number	Incapacitation Time	
			Actual (min:sec)	Mean \pm Standard Deviation (min:sec)
30.9	90	1	9:15	44:59 \pm 32:31
		2	19:45	
		3	21:00	
		4	62:45	
		5	71:50	
		6	86:20	
46.4	60	1	12:15	36:39 \pm 12:41
		2	33:50	
		3	40:40	
		4	42:30	
		5	44:40	
		6	46:00	

TABLE 21

Constants for Time-Concentration Hyperbolas ($Y = Q + R/X$)

Material	Laboratory	Flaming		Non-flaming		440°C	
		Q	R	Q	R	Q	R
ABS	1	3.2	168.0	-6.1	738.0	-13.8	1011.5
	5	12.6	56.7	0.7	492.5	d	
DFIR	1	13.3	231.4	12.8	226.9	H	
	2	5.0	389.2	17.7	99.0	H	
	3	7.2	263.8	7.3	107.0	H	
	6a	13.2	317.3	16.6	230.6	H	
	6b	13.8	246.2	18.3	98.7	H	
	8	9.3	230.3	10.3	242.7	H	
FPU	6a,b	-42.9	2739.0	G		H	
MOD	2	N.D.		-7.7	161.3	N.D.	
	5	-2.3	79.8	-0.3	62.9	4.5	78.4
	6	-2.8 ^a	82.9 ^a	-18.6 ^a	149.9 ^a	-3.4 ^{a,b}	157.4 ^{a,b}
PPS	2	10.5	259.8	7.4	212.0	C	
	4	e		17.11	203.9	C	
	5	12.1	92.5	9.2	99.8	N.D.	
PSTY	2	-25.3	1689.4	G		H	
	6b	-6.4	649.2	d		H	
PTFE	1	6.5	27.0	4.9	16.7	d	
PVC	3	e		2.8	161.4	C	
PVCZ	1	-3.8	347.9	2.1	78.6	6.5	130.0
	2	15.0	1.9	-51.7	888.9	d	
REDO	5	9.2	441.5	16.8	172.5	H	
	6	12.0 ^{a,b}	385.3 ^{a,b}	5.8 ^a	480.0 ^a	H	
	8	5.0	856.6	12.0	299.2	H	
RPU	8	4.5	139.3	-3.9	940.6	G	
WOOL	2	2.2	586.0	4.4	272.6	d	
	3	-10.9	517.0	e		e	
	6	5.7 ^a	409.9 ^a	-12.0 ^a	867.9 ^a	11.2 ^b	332.3 ^b
	8	11.0	497.1	-9.5	891.1	-61.9	2595.9

a: NBS small furnace

b: NBS large furnace

C: No data points

d: One data point

e: Two data points

G: No incapacitation

N.D. Not determined

H: Non-flaming temperature within 50°C of 440°C

TABLE 22
EC₅₀ Values¹⁹ (mg/l)

Material	Laboratory	Flaming	Non-flaming	440°C
ABS	1	10.6(7.4 - 15.2) ^{c*}	~21.0[15.1 ¹ - 25.2 ¹²] ^d	~20.2[15.1 ¹ - 25.2 ¹³]
	3	6.0(4.1 - 8.9)	5.8(2.8 - 8.4)	9.0(4.7 - 17.3)
	5	~17.0[15.0 ⁷ - 20.0 ¹³]	~23.0[18.5 ¹ - 27.5 ¹³]	<37.6 ¹³
DFIR	1	20.0(16.4 - 24.3)	15.0(12.3 - 18.2)	H
	2	18.4(14.0 - 24.1)	10.1(7.2 - 14.2)	H
	3	~14.5[10.0 ¹ - 19.1 ¹³]	5.6(3.1 - 9.9)	H
	4	N.D.	22.0(13.2 - 36.7)	H
	5	14.0(10.5 - 18.6)	19.2(14.3 - 25.8)	H
	6a	21.8(15.5 - 30.7) ¹⁷	18.3(14.5 - 23.0)	H
	6b	~23.5[23.0 ¹ - 24.0 ¹³]	13.5(12.9 - 14.2)	H
	8	~20.9 ^{3,13}	14.7(13.3 - 16.2)	H
FPU	3	9.6(4.1 - 22.1)	7.0(3.6 - 13.6)	H
	4	~49.5 ^{10,15}	20.2(8.6 - 47.3)	H
	6a,b	37.5(35.8 - 39.3)	53.0(40.1 - 69.9)	H
MOD	2	N.D.	2.7(2.1 - 3.4)	N.D.
	5	~2.8[2.0 ¹ - 3.0 ⁹⁺]	~3.0[2.0 ¹ - 4.0 ¹³]	~5.0[4.0 ¹ - 6.0 ¹³]
	6	3.1(2.2 - 4.3) ^{a,17}	3.2(2.8 - 3.7) ^a	6.4(5.8 - 7.0) ^{a,b}
PPS	2	<15 ¹²	8.8(6.8 - 11.2)	>19.9 ¹
	4	21.8(12.9 - 36.7)	19.0(10.2 - 35.3)	N.D.
	5	<10 ¹³	<7.0 ¹³	>40.0 ¹
PSTY	2	~30.0 ¹⁰	50.0 ¹	H
	6b	~28.7[27.5 ¹ - 30.4 ¹³]	>40.0 ¹	H
PTFE	1	~0.80[0.063 - 1.514 ¹³]	0.68(0.31 - 1.49)	~15.2[15.1 ¹ - 25.2 ¹³]
	6a	>0.25 ¹	>5.03 ¹	N.D.
PVC	3	6.0(4.0 - 8.9)	~9.4 ^{10,16}	13.5(4.9 - 36.8)
	6b	~18.5[17.5 ⁵ - 19.8 ¹³]	>30.0 ⁵	>30.0 ⁵
PVCZ	1	11.8[10.1 ⁸ - 15.1 ¹³]	~5.4[5.1 ¹ - 10.1 ¹³]	7.6(4.5 - 12.7)
	2	13.2(11.3 - 15.4)	11.7(10.3 - 13.2)	~12.4 ^{10,15}
REDO	5	<40.6 ¹³	<25.0 ¹³	H
	6	34.8(31.1 - 39.0) ^{a,b}	~23.0[22.5 ¹ - 24.2 ¹³] ^a	H
	8	51.0(46.1 - 56.5)	~24.1 ^{3,13}	H
RPU	8	8.9(5.1 - 15.6)	~29.3 ⁶ [29.3 ¹ - 35.1 ¹³]	>35.2 ¹
WOOL	2	23.8(16.0 - 35.3)	~17.0[15.0 ⁵ - 20.0 ¹³]	>27.0 ⁷
	3	~17.2[9.7 ⁸ - 19.0 ¹³]	6.8(4.2 - 11.1)	~23.3[19.3 ⁴ - 30.1 ¹³]
	6	~22.3[22.1 ¹ - 22.6 ¹³] ^a	19.7(16.2 - 24.0) ^a	24.5(23.0 - 26.1) ^b
	8	<45.0 ¹³	24.0(20.3 - 28.3)	~29.3 ⁶ [29.3 ¹ - 35.2 ¹³]

a: NBS small furnace

b: NBS large furnace

c: (95% confidence limits)

~: Estimated [values used to determine estimate]

H: 440°C is greater than or within 50°C of non-flaming temperatures

N.D. Not determined

1: 0% affected

Superscripts 2-12 refer to number of animals affected/number of animals tested

2: 1/2

3: 1/3

4: 1/5

5: 1/6

6: 2/3

7: 2/5

8: 2/6

9: 3/5

9+: 4/5

10: 3/6

11: 4/6

12: 5/6

13: 100% affected

14: No data points between 0% effect and 100% effect

15: One data point only

16: One data point between 0% effect and 100% effect

17: Significantly heterogeneous data

18: Late post-exposure deaths not counted

19: Litchfield, J.T. and Wilcoxon, F., reference 7

TABLE 23*

LC₅₀ (30 minutes + 14 days) in mg/l¹⁹

Material	Laboratory	Flaming	Non-flaming	440°C
ABS	1	15.0(12.3-18.3) ^c	19.3(13.9-26.9)	30.0(26.5-34.0) ¹⁸
	3	15.6(13.2-18.4)	>38.4 ⁵	>38.0 ⁸
	5	20.8(15.9-27.2)	33.3(23.1-47.9)	>37.6 ⁴
	6b	19.3(16.7-22.3)	30.9(21.2-45.0)	N.D.
DFIR	1	35.8(28.6-44.9) ¹⁸	16.7(14.5-19.3)	H
	2	45.3(39.0-52.7)	27.6(22.9-33.3)	H
	3	~24[19.0 ¹ - 29.0 ¹²] ¹⁶	26.8(21.3-33.7)	H
	4	29.6(22.7-38.6)	24.0(19.9-29.0)	H
	5	38.4(35.2-41.9)	25.9(20.0-33.5)	H
	6a	41.0(33.0-50.9)	20.4(16.4-25.3)	H
	6b	39.8(38.2-41.4)	22.8(20.2-25.8)	H
	8	29.8(23.9-37.1)	18.5(17.3-19.8)	H
FPU	3	>38.0 ¹	27.8(16.9-45.8)	H
	4	>49.5 ¹	40.0(31.2-51.3)	H
	6a&b	>40.0 ⁵	26.6(15.3-46.2) ¹⁷	H
MOD	2	N.D.	5.2(4.9-5.5)	N.D.
	4	7.1(6.4-7.9)	7.8(6.3-9.7)	10.0(6.9-14.4)
	5	4.7(3.2-6.9)	7.0(5.0-9.7)	~5.7[4 ¹ -6 ⁹] ¹⁶
	6	4.4(3.9-5.0) ^a	5.3(4.0-7.1) ^a	7.3(6.3-8.5) ^{a,b}
PPS	2	25.3(22.0-29.2)	18.7(15.2-23.0)	>19.9 ¹
	4	~36[24.9 ¹ -39.6 ¹¹] ¹⁶	32.2(27.7-37.5)	>9.9 ¹
	5	11.7(9.1-15.0)	10.7(8.4-13.6)	>40.0 ¹
	6b	19.8(14.8-26.5)	9.5(9.1-10.1)	N.D.
PSTY	2	53.5(41.4-69.1)	>50.0 ¹	H
	4	32.6(30.5-34.8)	>46.2 ¹	N.D.
	6b	38.9(37.9-39.9)	>40.0 ¹	H
PTFE	1	0.164(0.073-0.367)	0.125(0.083-0.188)	~15[5.0 ¹ -25.0 ¹³] ¹⁴
	4	0.400(0.02-6.81)	0.235(0.05-1.20)	N.D.
	6a	0.045(0.039-0.054)	0.045(0.017-0.120)	N.D.
PVC	3	~15[10 ¹ -19 ¹²] ¹⁶	~16[14 ⁸ -19 ¹³] ¹⁶	20.7(14.0-30.7)
	6b	17.3(14.8-20.2)	20.0(14.7-27.2)	25.0(20.2-31.0)
PVCZ	1	9.4(7.2-12.3)	7.6(5.5-10.5)	8.5(6.1-11.9)
	2	14.3(12.5-16.3)	13.3(11.5-15.4)	>12.4 ⁴
	6b	~15[15.0 ¹ -15.5 ¹³]	11.3(8.5-14.9)	12.8(12.1-13.6)
REDO	5	45.0(39.9-50.8)	25.0(18.7-35.5)	H
	6	56.8(51.6-62.5) ^{a,b}	30.3(26.0-35.4) ^a	H
	8	60.0(56.6-63.6)	35.0(24.5-50.1)	H
RFU	4	>38.4 ¹	>34.0 ¹	>39.6 ¹
	6b	13.3(12.2-14.5)	>39.6 ¹	N.D.
	8	11.3(7.6-16.8)	>35.1 ²	>35.2 ¹
WOOL	2	42.8(36.6-50.1)	25.2(18.4-34.6)	>27.2 ¹
	3	~23[19 ¹ - 24 ¹³] ¹⁴	15.8(13.5-18.6)	~25[19 ¹ - 30 ¹¹] ¹⁶
	6	28.2(23.0-34.5) ^a	25.1(22.3-28.3) ^a	32.1(30.2-34.1) ^b
	8	60.0(46.6-77.3)	28.5(23.5-34.6)	32.6(28.7-37.0)

*For explanation of superscript letters and numbers, see legend to table 22.

TABLE 24

Slopes of LC_{50} , 30 min. + 14 days (95% confidence limits of slope)¹⁹

Material	Laboratory	Flaming	Non-flaming	440°C
ABS	1	1.58(1.34-1.85) ²⁰	1.80(1.02-3.16)	1.20(1.07-1.36)
	3	1.23(1.08-1.40)	C	C
	5	1.46(1.13-1.89)	2.06(0.86-4.92)	C
	6b	1.37(1.09-1.72)	1.23(0.99-1.54)	N.D.
DFIR	1	1.41(1.06-1.89)	1.38(1.05-1.81)	H
	2	1.30(1.00-1.69)	1.26(1.04-1.53)	H
	3	E	1.64(0.0-3.85)	H
	4	1.69(0.66-4.30)	1.69(1.29-2.22)	H
	5	1.14(1.01-1.28)	1.73(1.25-2.41)	H
	6a	1.66(0.81-3.39)	1.43(1.09-1.88)	H
	6b	1.05(1.02-1.08)	1.25(1.10-1.42)	H
	8	1.51(0.61-3.73)	1.08(1.04-1.12)	H
FPU	3	C	1.87(1.19-2.94)	H
	4	C	2.37(1.46-3.84)	H
	6a&6b	C	2.37(0.93-6.01) ¹⁷	H
MOD	2	N.D.	1.11(1.02-1.21)	N.D.
	4	1.14(1.00-1.30)	1.40(1.18-1.66)	1.91(1.04-3.52)
	5	1.88(1.22-2.89)	1.70(1.23-2.36)	E
	6	1.30(1.10-1.53) ^a	1.67(0.99-2.81) ^a	1.35(1.09-1.68) ^{a, b}
PPS	2	1.23(1.09-1.38)	1.35(1.02-1.78)	C
	4	E	1.21(0.98-1.49)	C
	5	1.41(1.00-2.00)	1.31(1.07-1.61)	C
	6b	1.50(0.50-4.47)	1.07(1.04-1.11)	N.D.
PSTY	2	1.51(0.71-3.20)	C	H
	4	1.14(1.07-1.23)	C	N.D.
	6b	1.03(1.00-1.07)	C	H

TABLE 24 (Continued)

Material	Laboratory	Flaming	Non-flaming	440°C
PTFE	1	4.15(1.67-10.28)	2.07(1.29-3.31)	E
	4	5.10(1.80-14.43) ¹⁷	5.27(0.79-35.17) ¹⁷	N.D.
	6a	1.33(0.98-1.81)	7.94(1.28-49.31)	N.D.
PVC	3	E	E	1.63(1.18-2.26)
	6b	1.22(1.12-1.34)	1.47(1.03-2.09)	1.36(0.90-2.06)
PVCZ	1	1.51(1.19-1.91)	1.88(1.04-3.41)	1.52(1.19-1.93)
	2	1.24(1.08-1.43)	1.30(1.07-1.58)	C
	6b	E	1.42(0.65-3.1)	1.09(1.01-1.17)
REDO	5	1.30(1.00-1.70)	1.78(0.69-4.62)	H
	6a	1.15(1.07-1.24)	1.32(1.06-1.65)	H
	8	1.07(1.03-1.11)	2.29(0.49-10.69)	H
RPU	4	C	C	C
	6b	1.10(0.95-1.28)	C	N.D.
	8	1.83(0.15-21.68)	C	C
WOOL	2	1.39(0.97-1.99)	1.62(0.89-2.95)	C
	3	E	1.22(1.10-1.36)	E
	6	1.60(0.91-2.83) ^a	1.17(1.06-1.29) ^a	1.39(0.82-2.36) ^b
	8	1.86(0.83-4.14)	1.47(0.89-2.41)	1.15(0.87-1.52)

a: NBS small furnace

b: NBS large furnace

C: No slope as LC_{50} > highest concentration testedD: No slope as LC_{50} < lowest concentration testedE: LC_{50} estimated

H: 440°C is greater than or within 50°C of non-flaming temperature

N.D. Not determined

17: Significantly heterogeneous data

19: Litchfield and Wilcoxon - reference 7.

20: Units are $\frac{\% \text{ Lethality}}{\text{mg/l}}$

Table 25

Multiplication Factor Between Lowest and
Highest LC₅₀ Value for Each Material^a

Material	Flaming Mode	Non-Flaming Mode
ABS	1.4	1.7 ^b
DFIR	1.9	1.7
FPU	c.	1.5
MOD	1.6	1.5
PPS	3.1	3.4
PSTY	1.6	c.
PTFE	8.9	5.2
PVC	1.2	1.3
PVCZ	1.6	1.8
REDO	1.3	1.4
RPU	1.2 ^b	c.
WOOL	2.6	1.8

- a. Values calculated from LC₅₀ values presented in Table 23.
- b. Data listed as > (greater than) in Table 23 were not used.
- c. No value calculated as the LC₅₀ values in Table 23 were all higher than the highest concentration tested.

Table 26

NBS Control Blood Values for Fischer 344 Rats

Blood Parameters	Mean	Standard Deviation	Number of Values
Total Hemoglobin (g/100 mL)	14.8	1.2	180
Oxyhemoglobin (%)	94.1	1.8	180
Carboxyhemoglobin (%)	1.7	0.8	180
Methemoglobin (%)	0.7	0.3	180
Volume % Oxygen (mL/100 mL)	19.3	1.7	180
pH	7.44	0.06	173
Partial Pressure CO ₂ (mm Hg)	35.0	4.1	151
Partial Pressure O ₂ (mm Hg)	95.6	8.1	138

Table 27

Carbon Monoxide, Carboxyhemoglobin, and Hydrogen Cyanide Concentrations at LC₅₀ (30 minutes) and EC₅₀ (30 minutes).
Mean Carboxyhemoglobin Concentrations at Time of Incapacitation

Mat'l.	Mode	Deaths	Lab. #	Auto.- Ignit. Temp. (°C)	Animals Cannul.	LC ₅₀ 30 min. + 14 days (mg/l)	LC ₅₀ 30 min. (mg/l)	CO ^c (ppm)	COHb ^d (%)	HCN ^c (ppm)	EC ₅₀ (mg/l)	CO ^c (ppm)	COHb ^d (%)	HCN ^c (ppm)	Mean COHb at Time of Incap. ± Standard Deviation (n) (%)
ABS	F	W,P	1	525	-	15.0	17.4	900	23	130	10.6	550	<23	80	44.6 ± 11.1 (3)
			3	555	+	15.6	15.6	1500	60	180	6.0	590	26	70	10.9 ± 5.2 (6)
			5	500	+	20.8	20.8	1500	44	160	~17.0	1200	37		26.0 ± 5.8 (3)
			6b	575	+	19.3	22.1	1800	44		N.D.	N.D.	N.D.		N.D.
			1	525	-	19.3	22.0	330	ID	50	~21.0	320	ID	~50	ID
			3	555	+	>38.4	>38.0	>850	>22	>160	5.8	130	6	24	1.3 ± 0.5 (4)
DFIR	F	W	5	500	+	33.3	33.0	420	18		~23.0	290	18		7.6 ± 3.9 (5)
			6b	575	+	30.9	>32.5	>700	>28	>160	N.D.	N.D.	N.D.		N.D.
			1	465	-	35.8	35.0	3100	60		20.0	1800	41		49.2 ± 3.5 (6)
			2	465†	-	45.3	50.1	3400	ID	ID	18.4	1200	ID		57.2 ± 4.5 (5)
			3	475	+	~24.0	~24.9	2700	86		~14.5	1600	73		58.5 ± 29.1 (8)
			4	485†	S	29.6	30.6	2700	65		N.D.	N.D.	N.D.		N.D.
NF	F	W,P	5	440†	S	38.4	38.4	3600	81		14.0	1300	61		57.1 ± 15.4 (2)
			6a	465	+	41.0	45.0	3300	84		21.8	1600	70		60.4 ± 11.8 (9)
			6b	465	+	39.8	39.8	3400	83		~23.5	2000	75		75.1 ± 4.9 (7)
			8	510	-	29.8	30.0	3100	ID	ID	~20.9	2100	ID		75.0 ± 3.9 (9)
			1	465	-	16.7	21.7	3000	57		15.0	2100	46		46.7 ± 13.4 (11)
			2	465†	-	27.6	42.9	4900	ID	ID	10.1	1100	ID		57.6 (1)
FPU	F	W	3	475	+	26.8	37.3	3900	83		5.6	580	34		19.0 ± 21.7 (25)
			4	485†	S	24.0	24.9	3100	79		22.0	2800	N.D.		N.D.
			5	440†	S	25.9	>46.5	>4400	>75		19.2	1800	75		71.7 ± 4.5 (4)
			6a	465	+	20.4	34.8	3700	84		18.3	1900	77		77.4 ± 7.8 (4)
			6b	465	+	22.8	29.0	3400	86		13.5	1600	66		60.8 ± 11.4 (9)
			8	510	-	18.5	20.5	3400	83		14.7	2500	ID		79.4 ± 5.0 (6)
NF	F	W	3	395	+	>38.1	>38.1	>1200	>42	>32	9.6	300	11	8	2.2 ± 0.4 (3)
			4	385	-	>49.5	>49.5	>1200	>64		~49.5	1200	64		N.D.
			6a,b	370	+	>40.0	>40.0	>1000	>65	>22	37.5	900	44	21	52.7 ± 16.0 (2)
			3	395	+	27.8	>37.9	>2200	~65	>10	7.0	400	32	2	7.3 ± 4.7 (7)
			4	385	+	40.0	>50.9	>1800	>56		20.2	700	48		N.D.
			6a,b	370	+	26.6	>47.7	>1500	>62	>18	53.0	1600	62	20	11.8 ± 3.3 (2)

Table 27 (continued)

Matl.	Mode	Deaths	Lab. #	Auto.- Ignit. Temp. (°C)	Animals Cannul.	LC ₅₀ 30 min. + 14 days (mg/ℓ)	LC ₅₀ 30 min. (mg/ℓ)	CO ^c (ppm)	COHb ^d (%)	HCN ^c (ppm)	EC ₅₀ (mg/ℓ)	CO ^c (ppm)	COHb ^d (%)	HCN ^c (ppm)	Mean COHb at Time of Incap. + Standard Deviation (n) (%)
MOD	F	W,P	4	740	-	7.1	7.3	570	N.D.		N.D.	N.D.	N.D.		N.D.
			5	710	-	4.7	5.0	420	13		~2.8	230	ID		10.0 ± 4.8 (4)
			6a	725	+	4.4	5.0	460	25	210	3.1	280	16	130	10.9 ± 3.8 (7)
	NF	W	2	725	-	5.2	5.2	170	ID		2.7	90	ID		6.8 ± 4.3 (2)
			4	740	-	7.8	8.9	400	N.D.		N.D.	N.D.	N.D.		N.D.
PPS	F	W,P	5	710	-	7.0	~7.5	580	13		~3.0	230	19		6.1 ± 4.1 (5)
			6a	725	+	5.3	5.2	420	15	250	3.2	260	9	150	2.6 ± 1.7 (2)
			2	675†	-	25.3	50.0	3300	81		<15.0	<1000	N.D.		50.8 ± 3.6 (5)
			4	694	-	~36.0	~39.6	3600	81		21.8	2000	71		N.D.
			5	645	+	11.7	15.2	3000	83		<10.0	<2000	<77		72.8 ± 5.4 (5)
PSTY	NF	W	6b	650	+	19.8	20.0	3500	82		N.D.	N.D.	N.D.		N.D.
			2	675†	-	18.7	18.7	3100	88		8.8	1500	52		60.1 ± 2.5 (10)
			4	694	-	32.2	32.2	2400	73		19.0	1400	72		N.D.
			5	645	S	10.7	11.0	3800	82		<7.0	<2400	<70		73.1 ± 6.8 (5)
			6b	650	+	9.5	9.7	4500	84		N.D.	N.D.	N.D.		N.D.
PSTY	F	W	2	450†	-	53.5	53.5	2500	77		~30.0	1400	N.D.		51.3 ± 8.0 (5)
			4	550	-	32.6	33.0	2300	67		N.D.	N.D.	N.D.		N.D.
			6b	490	+	38.9	38.9	1300	78		~28.7	1000	66		47.2 ± 11.9 (9)
			2	450†	-	>50.0	>50.0	>100	>6		>50.0	>100	>6		N.D.
			4	550	-	>46.2	>46.2	>600	N.D.		N.D.	N.D.	N.D.		N.D.
PTFE	NF	ID	6b	490	+	>40.0	>40.0	>70	>6		>40.0	>70	>6		3.3 ± 1.5 (2)
			1	650	-	0.164	1.01	N.D.	N.D.		~0.80	N.D.	N.D.		N.D.
			4	685	S	0.400	2.6	N.D.	17.9*		N.D.	N.D.	N.D.		N.D.
			6a	620	+	0.045	>0.25	N.D.	>2.5		>0.25	N.D.	>2.5		N.D.
			1	650	-	0.125	0.9	N.D.	N.D.		0.68	N.D.	N.D.		2.5*
PTFE	NF	P	4	685	S	0.235	>0.99	N.D.	12.8*		N.D.	N.D.	N.D.		N.D.
			6a	620	+	0.045	>5.03	N.D.	>2.5		>5.03	N.D.	>2.5		2.4*

Table 27 (continued)

Matl.	Mode	Deaths	Lab. #	Auto.- Ignit. Temp. (°C)	Animals Cannul.	LC ₅₀ 30 min. + 14 days (mg/kg)	LC ₅₀ 30 min. (mg/kg)	CO ^c (ppm)	COHb ^d (%)	HCN ^c (ppm)	EC ₅₀ (mg/kg)	CO ^c (ppm)	COHb ^d (%)	HCN ^c (ppm)	Mean COHb at Time of Incap. + Standard Deviation (n) (%)
PVC	F	P	3	625	+	~15.0	>38.1	>2200	>56		6.0	340	16		7.6 ± 5.1 (5)
			6b	600	+	17.3	>30.0	>1900	>79		~18.5	1100	53		69.4 ± 5.7 (2)
NF	NF	W,P	3	625	+	~16.0	>28.5	>1100	>20		~9.4	360	15		3.3 ± 2.9 (4)
			6b	600	+	20.0	>25.0	>700	>35		~30.0	880	41		28.3 ± 13.0 (3)
PVCZ	F	W,P	1	625	-	9.4	13.4	700	N.D.	130	~11.8	610	N.D.	~114	20.5 ± 2.4 (3)
			2	625†	-	14.3	15.4	1100	50		13.2	950	50		27.2 ± 24.3 (2)
	NF	W,P	6b	675	+	~15.0	15.2	2200	68	120	N.D.	N.D.	N.D.		N.D.
			1	625	-	7.6	9.6	390	9	130	~5.4	220	9	~70	8.5 ± 4.9 (3)
			2	625†	-	13.3	15.3	710	29		11.7	540	29		7.9 ± 9.5 (4)
			6b	675	+	11.3	>14.0	>1400	>44	>190	N.D.	N.D.	N.D.		N.D.
REDO	F	W	5	465	-	45.0	45.3	2800	83		<40.6	<2700	<80		N.D.
			6a,b	480	+	56.8	59.0	2900	83		34.8	1700	74		58.4 ± 7.3 (5)
			8	510	-	60.0	~65.0	3300	78		51.0	2600	62		75.4 ± 3.9 (7)
NF	NF	P	5	465	-	25.0	40.0	3500	85		<25.0	<2200	<71		N.D.
			6a	480	+	30.3	>45.0	>3600	>81		~23.0	1900	64		52.7 ± 23.4 (9)
			8	510	-	35.0	35.2	3400	84		~24.1	2300	57		78.5 ± 3.0 (7)
RPU	F	W,P	4	594	-	>38.4	>38.4	>1200	>56		>38.4	>1200	N.D.		N.D.
			6b	550	+	13.3	14.3	1800	63	140	N.D.	N.D.	N.D.		N.D.
			8	615	-	11.3	14.4	1400	51		8.9	880	31		41.7 ± 8.1 (7)
NF	NF	ID	4	594	-	>34.0	>34.0	>600	>42		N.D.	N.D.	N.D.		N.D.
			6b	550	+	>39.6	>39.6	>1700	>47	>44	N.D.	N.D.	N.D.		N.D.
			8	615	-	>35.1	>35.1	>1400	ID		~29.3	1200	ID		58.0 ± 4.0 (3)

Table 27 (continued)

Mat.	Mode	Deaths	Lab. #	Auto.- Ignit. Temp. (°C)	Animals Cannul.	LC ₅₀ 30 min. + 14 days (mg/l)	LC ₅₀ 30 min. (mg/l)	CO ^c (ppm)	COHb ^d (Z)	HCM ^c (ppm)	EC ₅₀ (mg/l)	CO ^c (ppm)	COHb ^d (Z)	HCN ^c (ppm)	Mean COHb at Time of Incap. + Standard Deviation (n)
WOOL	F	W,P	2	660†	-	42.8	>50.0	>1100	>46	~250	23.8	520	>45		28.4 ± 7.0 (7)
			3	695	+	~23.0	~23.8	1600	57	~250	~17.2	1200	48	~180	18.2 ± 15.0 (5)
			6	650	+	28.2	40.9	1000	47	190	~22.3	550	28	~100	25.1 ± 3.5 (4)
			8	675	-	60.0	58.3	1400	57		<45.0	<1100	<57		44.5 ± 5.2 (8)
NF	W,P		2	660†	-	25.2	45.1	1200	ID	ID	~17.0	470	ID		21.0 ± 10.3 (7)
			3	695	+	15.8	15.8	1100	39	190	6.8	460	23	80	6.7 ± 2.9 (7)
			6	650	+	25.1	29.5	1100	41	280	19.7	720	39	190	24.2 ± 9.8 (8)
			8	675	-	28.5	29.1	900	26		24.0	710	26		18.9 ± 5.0 (6)

a: NRS small furnace.

b: NRS large furnace.

c: Average calculated for 30 minute exposure at LC₅₀ (30 minutes) or EC₅₀.

d: COHb values taken just before end of 30 minute exposure.

ID: Insufficient data.

n: Number of values used to calculate mean.

N.D.: Not determined.

P: Post-exposure deaths.

W: Within exposure deaths.

*: Highest value.

†: If a range of values were given for the auto-ignition temperature, the lowest one is used for this table.

+: Animals were cannulated.

-: Animals were not cannulated.

S: Animals were cannulated sometimes.

F: Flaming

N.F.: Non-flaming

Table 28

Distribution-free Sign Test on COHb Values at the
LC₅₀ (30 minutes + 14 days)

Material	Mode	COHb Values (%)		Lab #1	2	Laboratory Effect†			6a	6b	8
		Mean	± S.D. (n)			3	4	5			
ABS	F	42.3	± 15.2(4)	-19.3		17.7		1.7		-0.3	
	NF	22.5	± 6.4(2)					-4.5		4.5	
DFIR	F	76.3	± 10.8(6)	-15.3		8.7	-12.3	4.7	7.7	6.7	6.0
	NF	75.0	± 11.7(7)	-26.0		6.0	3.0	0	5.0	6.0	
FPU	NF	57.3	± 5.8(3)			6.7	-3.3		-3.3		
MOD	F	17.5	± 6.4(2)					-4.5	4.5		
	NF	15.0	± 1.4(2)					-1.0	1.0		
PPS	F	80.5	± 1.3(4)		0.5		-0.5	-1.5		1.5	
	NF	81.8	± 6.3(4)		6.2		-8.8	0.2		2.2	
PSTY	F	74.0	± 6.1(3)		3.0		-7.0			4.0	
PVC	F	45.0	± 5.7(2)			-4.0				4.0	
	NF	23.5	± 5.0(2)			-3.5				3.5	
PVCZ	F	59.0	± 12.7(2)		-9.0					9.0	
	NF	24.7	± 14.0(3)	-15.7	4.3					11.3	
REDO	F	79.7	± 5.8(3)					3.3	3.3		-6.7
	NF	78.0	± 6.2(3)					-7.0	2.0		5.0
RPU	F	50.5	± 14.8(2)							10.5	-10.5
WOOL	F	50.8	± 7.3(4)		-4.8	6.2			-7.8		6.2
	NF	35.3	± 8.1(3)			3.7			5.7		-9.3

Total Number of effects	4	6	8	6	10	9	12	6
Number Negative effects	4	2	2	5	5	2	1*	3
Number Positive effects	0	4	6	1	4	7	11	3
% Negative effects	100	33.3	25	83.3	50	22	8.3	50
% Positive effects	0	66.7	75	16.7	40	78	91.6	50
Number Minimum effects	4	1	2	2	5	1	0	3
Number Maximum effects	0	1	3	0	0	3	8	1
% Minimum effects	100	16.7	25	33.3	50	11	0	50
% Maximum effects	0	16.7	37.5	0	0	33.3	66.7	16.7

Mean: Mean of COHb values from all laboratories who tested material

S.D.: Standard deviation of mean of COHb values

(n): number of values used to calculate mean

†: laboratory values minus mean value

F: Flaming

NF: Non-flaming

a: NBS small furnace

b: NBS large furnace

*: indicates a statistically significant number of positive or negative effects at the 5% level, reference 14

Table 29

Distribution-free Sign Test on COHb Values at the LC₅₀ (30 minutes)

Material	Mode	COHb Values (%)		Lab #	1	2	3	Laboratory Effect†					6a	6b	8
		Mean	S.D.					(n)	4	5	6				
ABS	F	42.8	15.2	(4)	-19.8		17.2		1.2		1.2				
DFIR	F	76.5	11.1	(6)	-16.5		9.5	-11.5	4.5		7.5		6.5		
	NF	78.7	10.9	(6)	-21.7		4.3	0.3			5.3		7.3	4.3	
MOD	F	19.0	8.5	(2)					- 6.0		6.0				
	NF	14.0	1.4	(2)					- 1.0		1.0				
PPS	F	81.2	1.0	(4)		- 0.2		- 0.2	1.8				0.8		
	NF	81.8	6.3	(4)		6.2		- 8.8	0.2				0.2		
PSTY	F	74.0	6.1	(3)		3.0		- 7.0					4.0		
PVCZ	F	59.0	12.7	(2)		- 9.0							9.0		
	NF	19.0	14.1	(2)	-10.0								10.0		
REDO	F	81.3	2.9	(3)					1.7		1.7			- 3.3	
	NF	84.5	0.7	(2)					0.5					- 0.5	
RPU	F	57.0	8.5	(2)									6.0	- 6.0	
WOOL	F	53.7	5.8	(3)			3.3				- 6.7			3.3	
	NF	35.3	8.1	(3)			3.7				5.7			- 9.3	

†For legend, see Table 28.

Table 30

Distribution-free Sign Test on COHb Values at EC₅₀

Material	Mode	COHb Values (%)		Lab #	1	2	3	4	5	6a	6b	8
		Mean	S.D.									
ABS	F	31.5	7.8	(2)			- 5.5		5.5			
	NF	12.0	8.5	(2)			- 6.0		6.0			
DFIR	F	64.0	13.9	(5)	-23.0		9.0		- 3.0	6.0	11.0	
	NF	59.6	18.8	(5)	-13.6		-25.6		15.4	17.4	6.4	
FPU	F	39.7	26.8	(3)			-28.7	24.3	4.3			
	NF	47.3	15.0	(3)			-15.3	0.7	14.7			
MOD	NF	14.0	7.1	(2)					5.0	- 5.0		
PPS	NF	62.0	14.1	(2)		-10.0		10.0				
PVC	F	34.5	26.1	(2)			-18.5				18.5	
	NF	28.0	18.4	(2)			-13.0				13.0	
PVCZ	NF	19.0	14.1	(2)	-10.0	10.0						
REDO	F	68.0	8.5	(2)					6.0			- 6.0
	NF	60.5	4.9	(2)					3.5			- 3.5
WOOL	F	38.0	14.1	(2)			10.0		-10.0			
	NF	29.3	8.5	(3)			- 6.3		9.7			- 3.3

Total number of effects

3

2

10

3

5

9

4

3

Number negative effects

3

1

8

0

1

2

0

3

0

0

Number positive effects

0

1

2

3

4

7

4

0

0

0

Percent negative effects

100

50

80

0

20

22

0

100

0

0

Percent positive effects

0

50

20

100

80

78

100

0

0

0

Number minimum effects

2

1

8

1

0

2

0

2

2

2

Number maximum effects

0

1

1

2

3

5

3

0

0

0

Percent minimum effects

67

50

80

33

0

22

0

67

0

0

Percent maximum effects

0

50

10

67

60

56

75

0

0

0

†For legend, see Table 28.

Table 31

Distribution-free Sign Test on COHb Values at Time of Incapacitation

Material	Mode	COHb Values (%)			Laboratory Effect†								
		Mean	S.D.	(n)	Lab #	1	2	3	4	5	6a	6b	8
ABS	F	27.2	16.9	(3)		17.4		-16.3		- 1.2			
	NF	4.5	4.5	(2)				- 3.2		3.1			
DFIR	F	61.8	9.7	(7)		-12.6	- 4.6	- 3.3		- 4.7	- 1.4	13.3	13.2
	NF	58.9	21.1	(7)		-12.2	- 1.3	-39.9		12.8	18.5	1.9	20.5
FPU	F	27.5	35.7	(2)				-25.3			25.2		
	NF	9.6	3.2	(2)				- 2.3			2.2		
MOD	F	10.5	0.6	(2)						- 0.5	0.4		
	NF	5.2	2.3	(3)			1.6			0.9	-2.6		
PPS	F	61.8	15.6	(2)			-11.0			11.0			
	NF	66.6	9.2	(2)			- 6.5			6.5			
PSTY	F	49.3	2.9	(2)			2.0					-2.1	
PVC	F	38.5	43.7	(2)				-30.9				30.9	
	NF	15.8	17.7	(2)				-12.5				12.5	
PVCZ	F	23.9	4.7	(2)		- 3.4	3.3						
	NF	8.2	0.4	(2)		0.3	- 0.3						
REDO	F	66.9	12.0	(2)							- 8.5		8.5
	NF	65.6	18.2	(2)							-12.9		12.9
WOOL	F	29.1	11.1	(4)			-0.7	-10.9			- 4.0		15.4
	NF	17.7	7.7	(4)			3.3	-11.0			6.5		1.2
Total number of effects						5	10	10	-	8	10	5	6
Number negative effects						3	6	10*	-	3	5	1	0*
Number positive effects						2	4	0	-	5	5	4	6*
Percent negative effects						60	60	100	-	38	50	20	0
Percent positive effects						40	40	0	-	63	50	80	100
Number minimum effects						2	3	9	-	1	3	1	0
Number maximum effects						2	3	0	-	3	4	3	4
Percent minimum effects						40	30	90		13	30	20	0
Percent maximum effects						40	30	0		38	40	60	67

†For legend, see Table 28.

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5. AUTHOR(S) Barbara C. Levin, Maya Paabo and Merritt M. Birky			
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11. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here) <p>Seven laboratories selected from academia, industry, and government evaluated the 1980 version of the NBS test method for assessing the toxicity of combustion products to determine the operability of the procedure and the reproducibility of results across laboratories. The experimental design specified that each laboratory was responsible for testing Douglas fir and three other materials from a total of twelve natural and synthetic materials. All laboratories were required to use similar exposure and combustion systems, to measure the autoignition temperatures of their materials, to determine the toxicity of the gaseous products released by the materials under both flaming and non-flaming conditions, to monitor chamber environmental conditions (temperatures, and oxygen, carbon monoxide, and carbon dioxide concentrations), and to measure blood carboxyhemoglobin in the test animals (rats). In addition, a few laboratories measured hydrogen cyanide generated from nitrogen-containing materials. Toxicity was evaluated on the basis of incapacitation (hind-leg flexion behavioral avoidance response) during the 30 minute exposure and of lethality during the exposure and 14 day post-exposure observation period. The results of this interlaboratory evaluation were statistically analyzed and, in most cases, demonstrated reproducible results across laboratories. Possible reasons for any inconsistencies are discussed. Sensitive experimental factors are identified and modifications to the test method which resulted from the experimental data collected during the interlaboratory evaluation are described.</p>			
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